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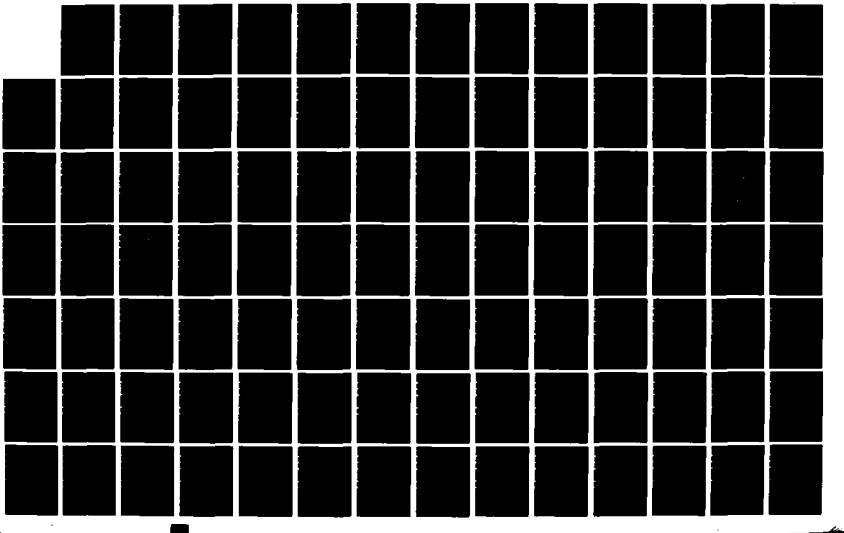

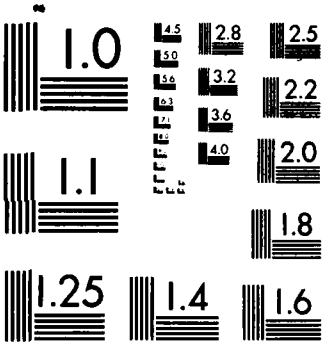

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Structural Chemistry of Energetic Compounds

ONR Annual Report

Contract No. N00014-85-WR-24060

1 October 1984 - 1 October 1985

Richard Gilardi, Clifford George and J. L. Flippen-Anderson

Laboratory for the Structure of Matter

Naval Research Laboratory, Washington, D.C. 20375

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X-ray diffraction, tetraazabicyclooctanes, oxetane, nitramines, nitroadamantane, Nitro radicals, Benzyl radicals, Tetrazoles, Cyclic compounds

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Structural characterization of 16 energetic materials and precursors was performed by x-ray diffraction. The materials examined include:

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(a) 1-Nitroso-2,2-di(trifluoromethyl)-1,3-diazacyclopentan-4-one
(b) 3,3,7,7,-Tetra(trifluoromethyl)-2,4,6,8-tetraazabicyclo [3.3.0]octane
(c) 2,6-Dinitro-3,3,7,7-tetra(trifluoromethyl)-2,4,6,8-tetraazabicyclo [3.3.0]octane
(d) 2,4,6-Trinitro-3,3,7,7-tetra(trifluoromethyl)-2,4,6,8-tetraaza-bicyclo[3.3.0]octane
(e) 2,4,6,8-Tetranitro-3,3,7,7,-tetra(trifluoromethyl)-2,4,6,8-tetra-azabicyclo[3.3.0]octane
(f) 3-Benzamido-6,8-dibenzoyl-1,3,5,6,8-pentaazabicyclo[3.2.2]nonane
(g) 1,3,5-Tribenzamido-1,3,5-hexahydrotriazine monohydrate
(h) 1-Benzoyl-4-benzamido-Δ^2-1,2,4-triazoline
(i) 2,7-Dibenzoyl-4-benzamido-1,2,4,6,7-pentaazabicyclo[1.2.4]nonane
(j) bis-Tetrazolo[1,5-a:1',5'-c]pyrazine
(k) 1,4-Dinitro-2,3,5,6-tetrabromo-2,3,5,6-piperazine
(l) 1,9-Diacetyl-3,5,7-trinitro-1,3,5,7-tetraaza-9-oxanonane
(m) 1,9-Diacetyl-3,5,7-trinitro-1,3,5,7-9-pentaazanonane
(n) 2-Oxa-6,9-diaza-6,9-dinitrospiro[3.6]decane
(o) 1-Aza-3,5,7-trinitroadamantane
(p) 7-Nitro-5,6-dihydro-7H-imidazolo[1,2-d]tetrazole

→ The trinitro and tetranitro tetraazabicyclo[3.3.0]octanes examined, compounds D and E, are significantly more dense than had been predicted. The crystal structure analysis showed that the increase in density is due to intramolecular crowding rather than extraordinary packing efficiencies which were at normal values of 0.73 and 0.75 respectively.

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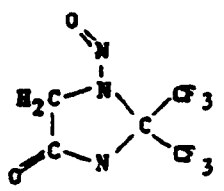

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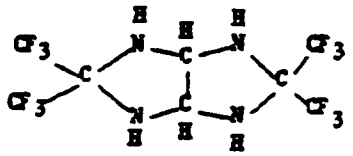

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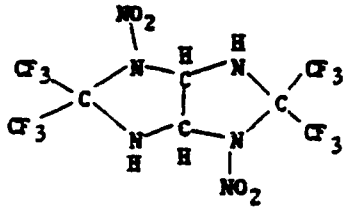

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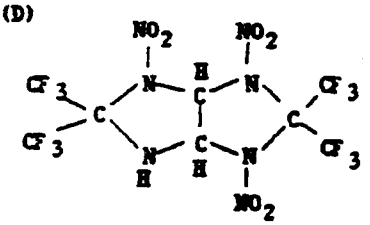

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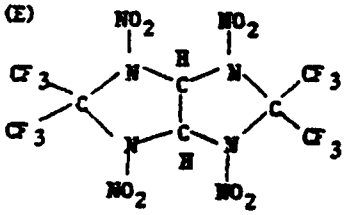

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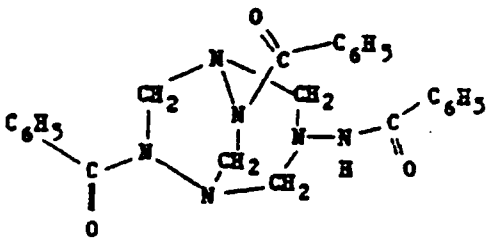

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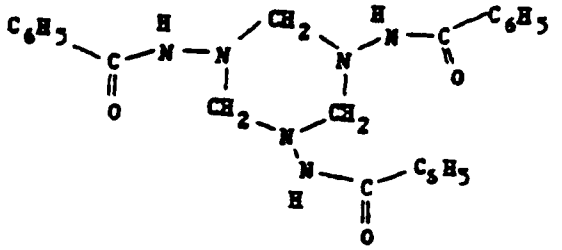

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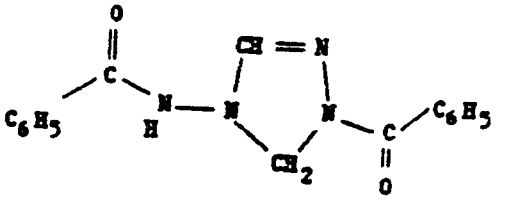

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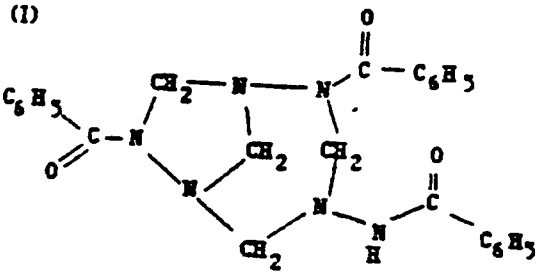

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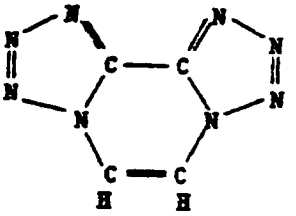

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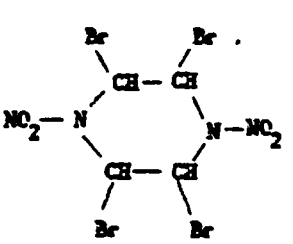

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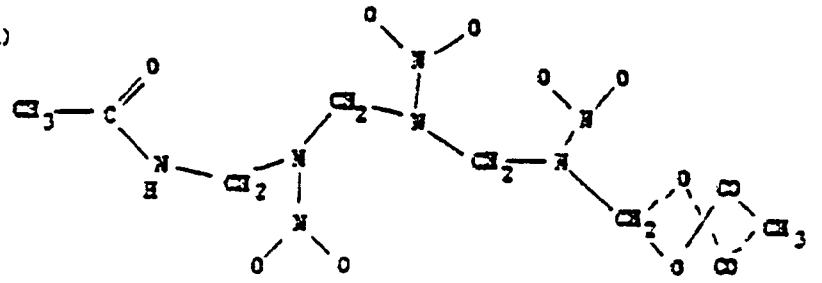

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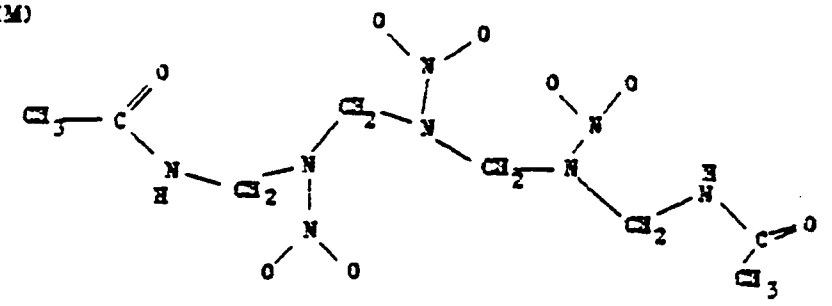

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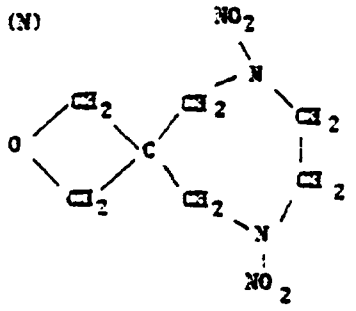

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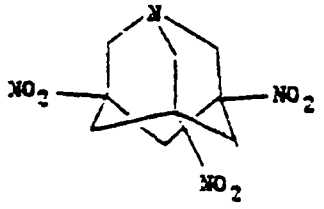

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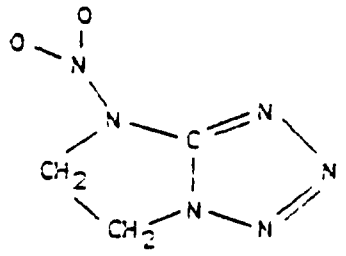


Synopsis of Results.

Molecules A through E and J and K were obtained from the Naval Surface Weapons Center (NSWC). Of these A, J, and K are either unexpected reaction products or synthetic intermediates which were produced in their energetic synthesis program. Although these three compounds are not themselves energetic materials, the identification of their structures and stereochemistry may aid in understanding the mechanisms of the reactions involved, and thus indirectly further the ONR synthesis program.

The series of tetraazabicyclooctanes, molecules B through E, are very interesting molecules closely related to bicyclo-HMX. In fact, compound E is bicyclo-HMX substituted with four trifluoromethyl groups; for certain applications, E may prove to be a useful explosive or fuel without further modification.

These four compounds are dense; each of the molecules has a density greater than the stable form of HMX. The crystallographic densities are 2.03, 1.98, 2.11, and 2.18 gm/cm^3 for B through E respectively. The densities of D and E are anomalously high with respect to densities derived from Holdens (NSWC) density prediction program, 2.02 and 2.04 gm/cm^3 respectively. Examination of the x-ray results show the source of the greater than expected density is intra-molecular crowding, not unusually close packing. All of the intermolecular contacts are greater than the usual close-packing limit (the sum of the van der Waals' radii of the atoms), except for a rather weak $NH\cdots NO_2$ hydrogen bond in D. The packing efficiency, which is the actual volume of the molecules in a cell divided by the volume of the cell, is near 0.75 for most crystals. For molecules

B through E, the packing efficiencies are 0.73, 0.68, 0.73, and 0.75, which are normal to low values.

The particular contacts which show severe crowding are the nitro-CF_3 contacts. This crowding also produces a number of distortions in the molecules (see table below). As one adds nitro groups to the parent compound, the bonds to the nitro and CF_3 groups lengthen and the N-C-N angles sharpen; in addition, the CF_3 group torsions deviate by almost 30° from a staggered configuration. All of these effects serve to lengthen the non-bonded CF_3-nitro distances; however, in compound E there are still 12 contacts which are extremely short (more than 0.1Å below normal van der Waals separations).

Selected Bond Distances, Angles, and Torsions

	Zero-NO_2	Di-NO_2	Tri-NO_2	Tetra-NO_2
C-CF_3(Å)	1.529	1.544	1.575	1.582
N-NO_2(Å)	-	1.386	1.405	1.413
N-C-N	105.6°	99.5°	99.7°	98.5°
C-C-C-F	177.3°	173.2°	161.7°	152.0°
N-O(Å)	-	1.217	1.206	1.207
C-F(Å)	1.324	1.298	1.310	1.323
Ring Torsions	14.0°	13.9°	12.2°	20.7°

The strain caused by short internal contacts makes the synthetic chemistry of bicyclo-HMX and its derivatives very difficult and unpredictable; however, this internal strain adds to its total energy content, and makes it an even more desirable energetic target compound.

Molecules F, G, H and I were synthesized by Drs. J. Boyer and G. Kumar of the University of Illinois at Chicago, in a systematic study

of the heterocyclic compounds produced by hydrazine/formaldehyde/orthoformate condensation and rearrangement reactions. The specific aim of this program of study is the synthesis of a "tri-hydrazine" analog of adamantane [2,3,5,7,8,9-hexaazatricyclo(3.3.1.$1^{3,7}$)decane]. This compound, if derivatized with nitro or nitramino groups, is expected to be a dense (d>2.1) explosive. Two new hetero-N cage compounds (F and I) were produced in FY85 by Boyer and Kumar and their structures were characterized by our x-ray diffraction analyses; energetic substitution has not yet been accomplished. Compound G is a precursor heterocycle from which F and I were made; compound H is a minor, lower molecular weight product formed in the same reaction which produces I. Crystals of both H & I were found in the crystalline reaction product.

Molecules L, M, and N were provided by C. Coon of Lawrence Livermore National Laboratories. Compounds L and M are energetic precursor molecules whose crystal structures were done primarily to corroborate their structural formulas. Compound N is a new energetic monomer, similar to 3,3-bis(methylnitroaminomethyl)oxetane, (BMNAMO). BMNAMO can be polymerized to form stable energetic homo- or co-polymers.

Molecule (O) is a novel monoazaadamantane synthesized by A. Nielsen of the Naval Weapons Center (China Lake). Of the four tertiary carbons in the adamantane cage, three are nitro-substituted and the other is replaced by a nitrogen atom. It is an energetic material, but its density (1.604 mg mm^{-1}) is much less than HMX; further nitro-substitution is theoretically possible but is considered to be unlikely.

Compound P was supplied by R. L. Willer of Morton Thiokol. This energetic molecule has the property of decomposing without going

through a melt phase.

The remainder of this report is a series of brief technical reports on each individual molecule and the x-ray experiment conditions. Fractional coordinates are provided for those intending further computational study; these are 'crystallographic' coordinates and must be converted to Cartesian Angstrom coordinates for some programs. Contact the authors if this creates a problem. Bond distances and angles are provided for all molecules; if specific torsion angles or non-bonded distances are required, they are on file at the Naval Research Laboratory (NRL) and available upon request.

1-Nitroso-2,2-di(trifluoromethyl)-1,3-diazacyclopentan-4-one,

$C_5H_3N_3O_2F_6$

Abstract

M_r = 251.088, orthorhombic, $Pna2_1$, $\underline{a}$ = 10.214(2), $\underline{b}$ = 10.986(3), $\underline{c}$ = 7.614(2)Å, V = 854.4(3)Å^3, Z = 4, D_x = 1.952 mg mm^{-3}, CuKα, λ = 1.54178Å, μ = 2.12 mm^{-1}, F_{000} = 496, T = 295K, R = .0511 and R_w = .0609 for 524 observed reflections, S = 1.295. Crystals volatile, sublime completely in a day; rapid data collection used to obtain unique set in three hours.

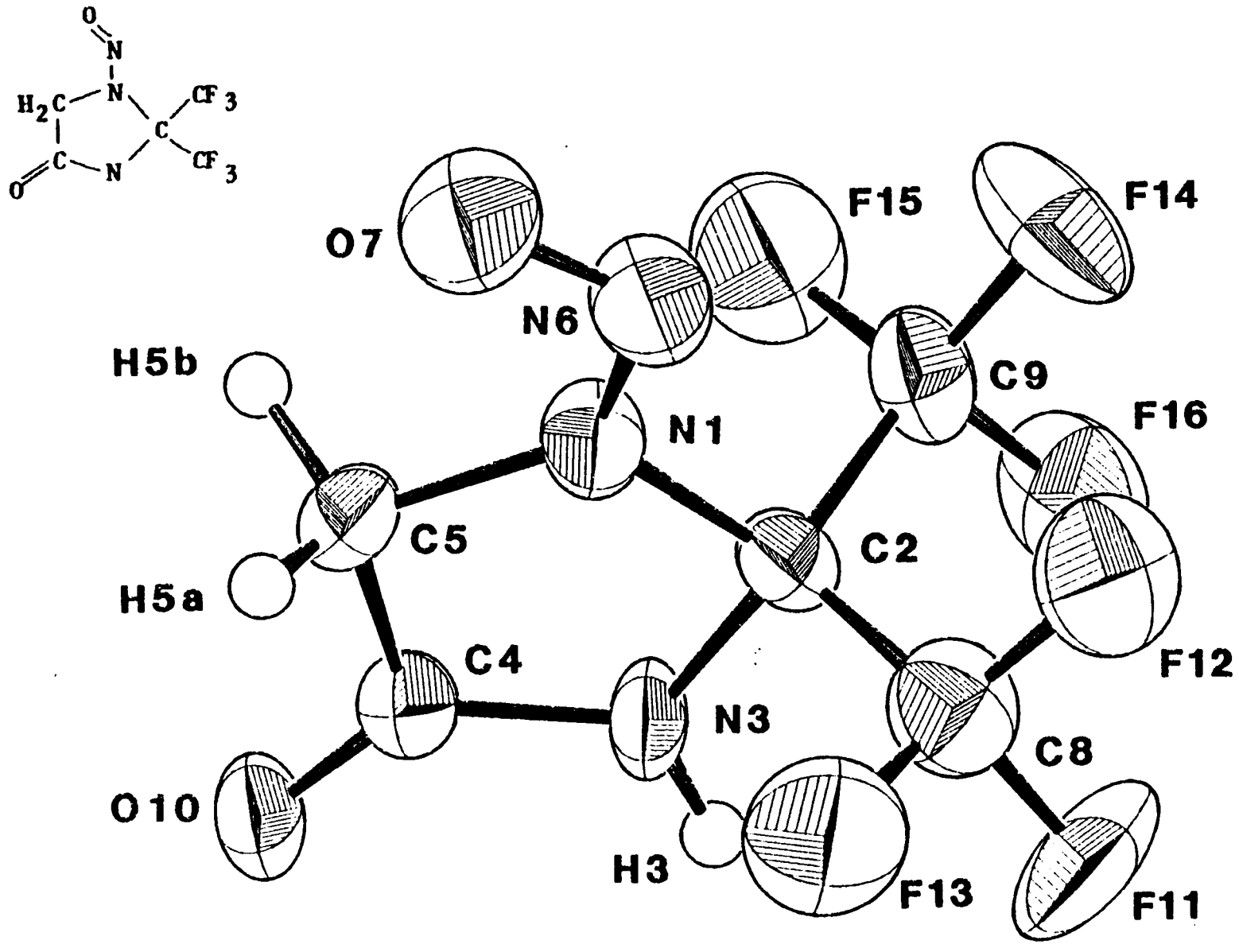
Experimental

Clear, colorless needle-shaped crystals provided by W. Koppes of the Naval Surface Weapons Center (Silver Spring, MD), grown by sublimation at 90°C/200 mm Hg. Data crystal (0.10 x 0.20 x 0.70 mm) centered on 25 reflections with $33° < 2\theta < 67°$ to obtain refined lattice parameters. Nicolet R3m diffractometer used, incident-beam graphite monochromator, $\theta/2\theta$ scans, scan width = $(1.8 + \Delta_{\alpha 1 \alpha 2})°$, $2\theta_{max}$ = 110°. In view of volatility of crystals and scarcity of material, rapid data collection used: scan rate = 30°/min for all reflections (3 hours for 1 unique set of data). First data set ($-10 \le h \le 0$, $0 \le k \le 11$, $0 \le \ell \le 8$) consisted of 696 observations, of which 36 were monitors, 524 were unique $F_o > 3\sigma(F_o)$ and 59 unique $F_o < 3\sigma(F_o)$. Monitor reflections (004, 620, and 040; measured after every 60 reflections) decreased uniformly from 100 to 87% of their original values (with individual variations from the mean of ±4%) over the course of 3 hours collection. Smoothed curve of monitor decrements used to correct all data. Second octant ($+h,+k,+\ell$) collected subsequently, monitors decreasing from 87% to 80% of original values;

numerous peak profiles off-center in second octant; second set discarded to avoid introduction of systematic errors.

Lorentz and polarization, but not absorption, corrections made. Structure solved using direct methods in $Pna2_1$ and Pnam (centric). Fifteen atoms in first $Pna2_1$ E-map; one more appeared with tangent-formula recycling. Since this result was an unexpected reaction product, atoms were identified by assigning C scattering factors to all atoms and refining occupancies of atoms, holding isotropic U_i's constant. All nitrogen and oxygen occupancies refined to 1.20-1.40 range, while carbon atom occupancies remained in 1.0-1.15 range. Eventual appearance of all hydrogen atoms in difference map corroborated atomic assignment.

Refinement: 150 parameters refined, all non-H atoms anisotropic, amino hydrogen refined on position and isotropic T factor, methylene hydrogens fixed to ride on carbon atoms with isotropic U's and tetrahedral geometry. R = 0.0511, Rw = 0.0609, S = 1.295 for 524 $Fo>3\sigma(Fo)$. Function minimized, $\Sigma w(F_o-Fc)^2$, where $w = [\sigma^2(F_o)+0.0009F_o^2]^{-2}$. Maximum $\Delta/\sigma = 0.040$, final difference map ripple extrema, 0.23 and $-0.25 eA^{-3}$.



1-Nitroso-2,2-di(trifluoromethyl)-1,3-diazacyclopentan-4-one

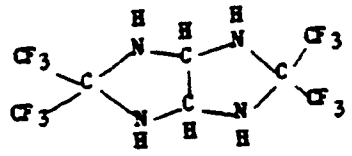
3,3,7,7-Tetra(trifluoromethyl)-2,4,6,8-tetraazabicyclo[3.3.0]octane

Abstract

$C_8H_6N_4F_{12}$, M_r = 386.14, monoclinic, P2/n, a = 10.431(4), b = 7.050(2), c = 25.778(14)Å, and β = 90.80(4). M.P. = 94 C°. V = 1895.7(13)Å^3, Z = 6, D_x = 2.029 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 2.336 mm^{-1}, F(000) = 1140, T = 295K, Final R = 0.053, wR = 0.056 for 2294 independent observed reflections. There are one and a half molecules in the asymmetric unit.

Experimental

Clear 0.35 x 0.22 x 0.15 mm. crystal crystallized from CH_2Cl_2 at 5 C°, Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 30<2θ<60 used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.59Å^{-1}, range of hkl: 0<h<11, 0<k<8, -30<l<30. Standards 600, 020, 004, monitored every 60 reflections with linear decrease of 8.9% during data collection, θ/2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (8°/min. minimum, 30°/min. maximum), 3601 reflections measured, 3112 unique, R_{int} = 0.025, 2294 observed Fo>3σ(Fo). Data corrected for Lorentz, polarization and absorption effects, and for the linear decrease in monitored intensities. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00050, 361 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms. (Δ/σ) max = -.16, R = 0.053, wR = 0.056, S = 1.510. Final difference Fourier excursions 0.26 and -0.32eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



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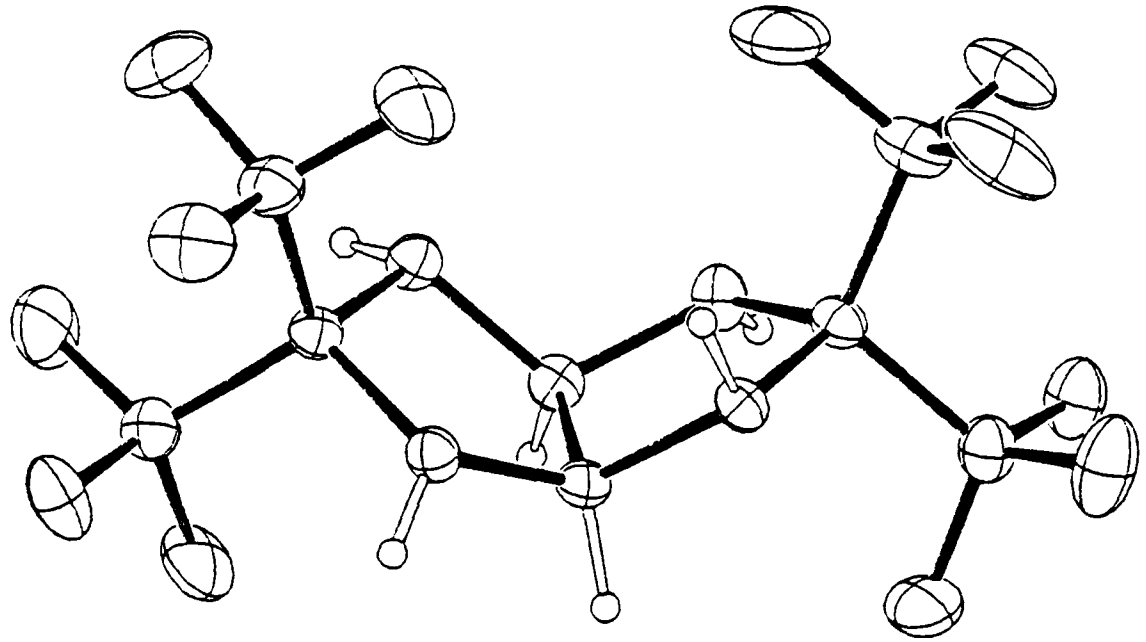
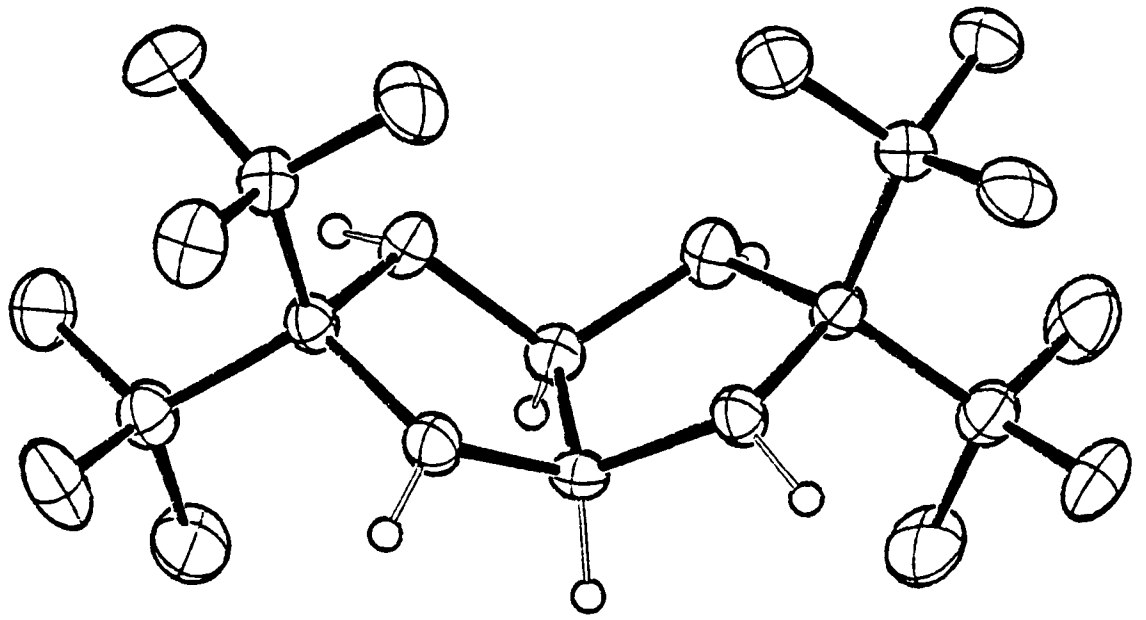




3,3,7,7-Tetra(trifluoromethyl)-

2,4,6,8-tetraazabicyclo(3.3.0)octane

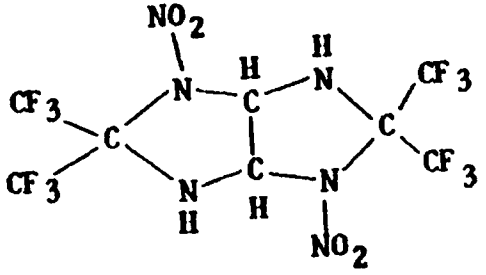
2,6-Dinitro-3,3,7,7-tetra(trifluoromethyl)-2.4,6,8-tetraazabicyclo[3.3.0]octane

Abstract

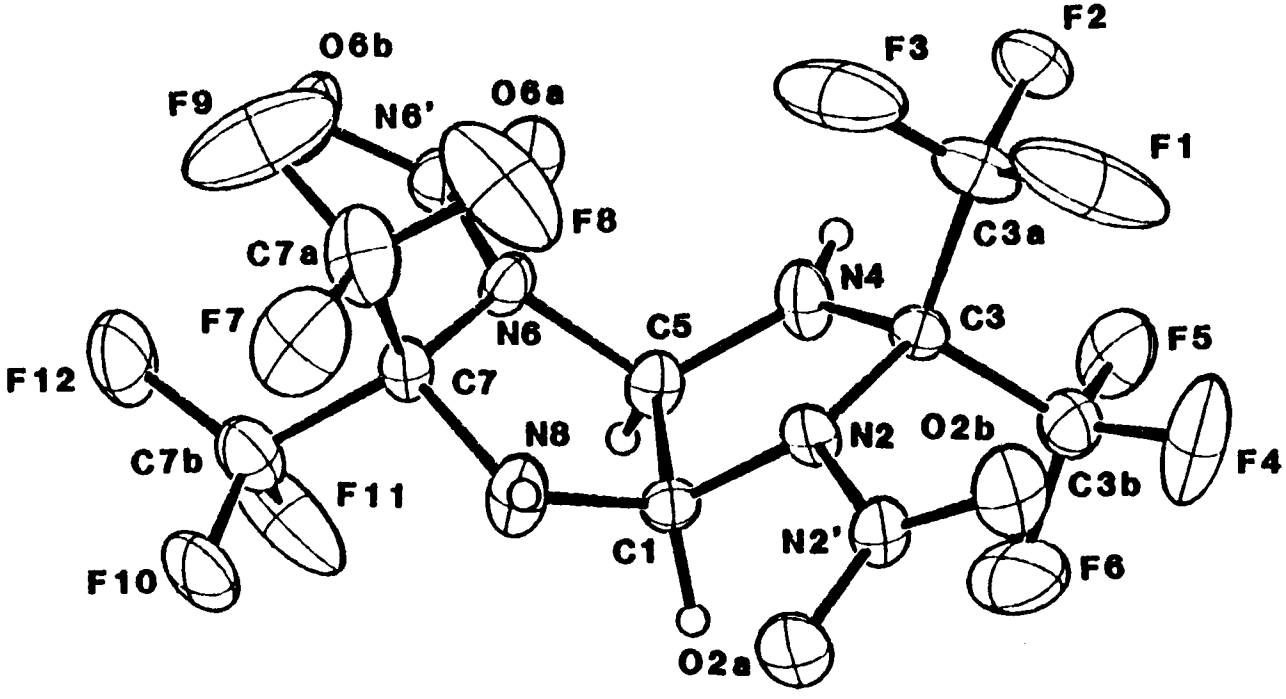
$C_8H_4N_6O_4F_{12}$, M_r = 476.14, orthorhombic, $Pna2_1$, a = 20.073(4), b = 7.353(1), c = 10.832(2)Å, V = 1598.7(4)Å^3, Z = 4, D_x = 1.978 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 2.22 mm^{-1}, F(000) = 936, T = 295K, Final R = 0.064, wR = 0.089 for 1205 independent observed reflections.

Experimental

Clear 0.10x0.15x0.65 mm. crystal, crystallized from CH_2Cl_2/hexane by W. Koppes of Naval Surface Weapons Center (Silver Springs, Md.). Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 40<2θ<60 used for determining lattice parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $(\sin\theta/\lambda)_{max}$ = 0.56Å^{-1}, range of hkl: -22<h<14, -8<k<0, 12<l<0 . Standards 004, 800, 020, monitored every 60 reflections with linear variation 4.1% over data collection, θ-2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (6°/min. minimum, 30°/min. maximum), 2393 reflections measured, 1263 unique, R_{int} = 0.021, 1205 observed Fo>3σ(Fo). Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00030 , 283 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, hydrogen isotropic temperature factors fixed at 1.2 U_{eq}(C). $(\Delta/\sigma)max$ = -0.012 R = 0.064, wR = 0.089, S = 3.65. Final difference Fourier excursions 0.34 and -0.28eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



(C)



2,6-Dinitro-3,3,7,7-tetra(trifluoromethyl)-

2,4,6,8-tetraazabicyclo(3.3.0)octane

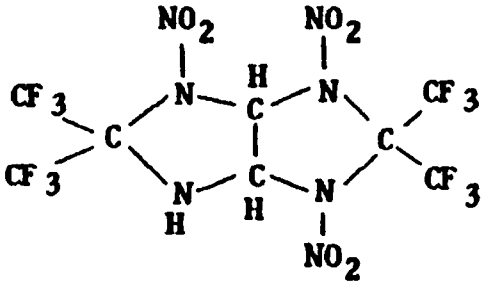
2,4,6-Trinitro-3,3,7,7-tetra(trifluoromethyl)-
2,4,6,8-tetraazabicyclo[3.3.0]octane

Abstract

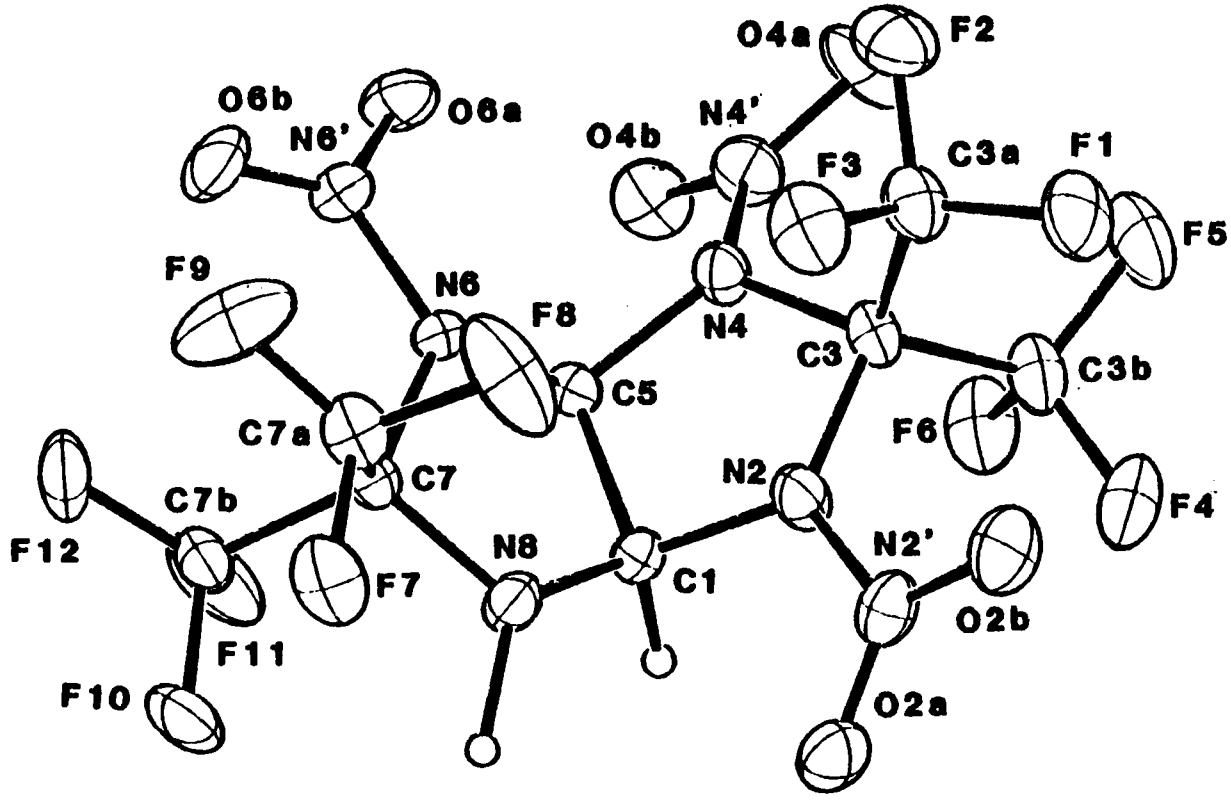
$C_8H_3N_7O_6F_{12}$, M_r = 521.13, monoclinic, $P2_1/c$, a = 6.390(6), b = 19.090(2), c = 12.347(1)Å, β = 95.38(1)°, V = 1640.1(3)Å^3, Z = 4, D_x = 2.110 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 2.35 mm^{-1}, F(000) = 1024, T = 295K, Final R = 0.049, wR = 0.074 for independent 2168 observed reflections.

Experimental

Clear 0.62x0.23x0.35 mm. crystal, crystallized from CH_2Cl_2 /hexane by W. Koppes of Naval Surface Weapons Center (Silver Springs, Md) Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 30<2θ<60 used for determining lattice parameters. Data corrected for Lorentz, polarization, and absorption effects, $(\sin\theta/\lambda)_{max}$ = 0.55Å^{-1}, range of hkl: -7<h<0, 0<k<19, -13<l<12. Standards 200,0 12 0, 006, monitored every 60 reflections with linear variation 8.3% over data collection, θ-2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate, (5°/min. minimum, 30°/min. maximum). 2568 reflections measured, 2259 unique, R_{int} = 0.023, 2168 observed Fo>3σ(Fo). Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00023 , isotropic secondary extinction value, 0.0019, 367 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms. $(\Delta/\sigma)max$ = 0.24, R = 0.049, wR = 0.074, S = 2.77. Final difference Fourier excursions 0.27 and -0.19eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



(D)



2,4,6-Trinitro-3,3,7,7-tetra(trifluoromethyl)-2,4,6,8-tetraazabicyclo(3.3.0)octane

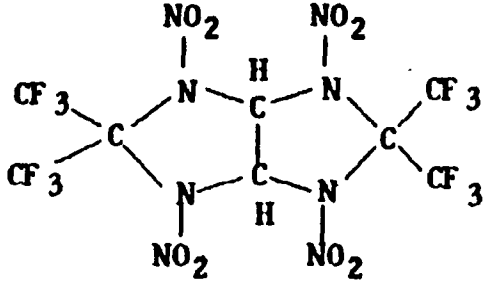
2,4,6,8-Tetranitro-3,3,7,7-tetra(trifluoromethyl)-2,4,6,8-tetraazabicyclo[3.3.0]octane

Abstract

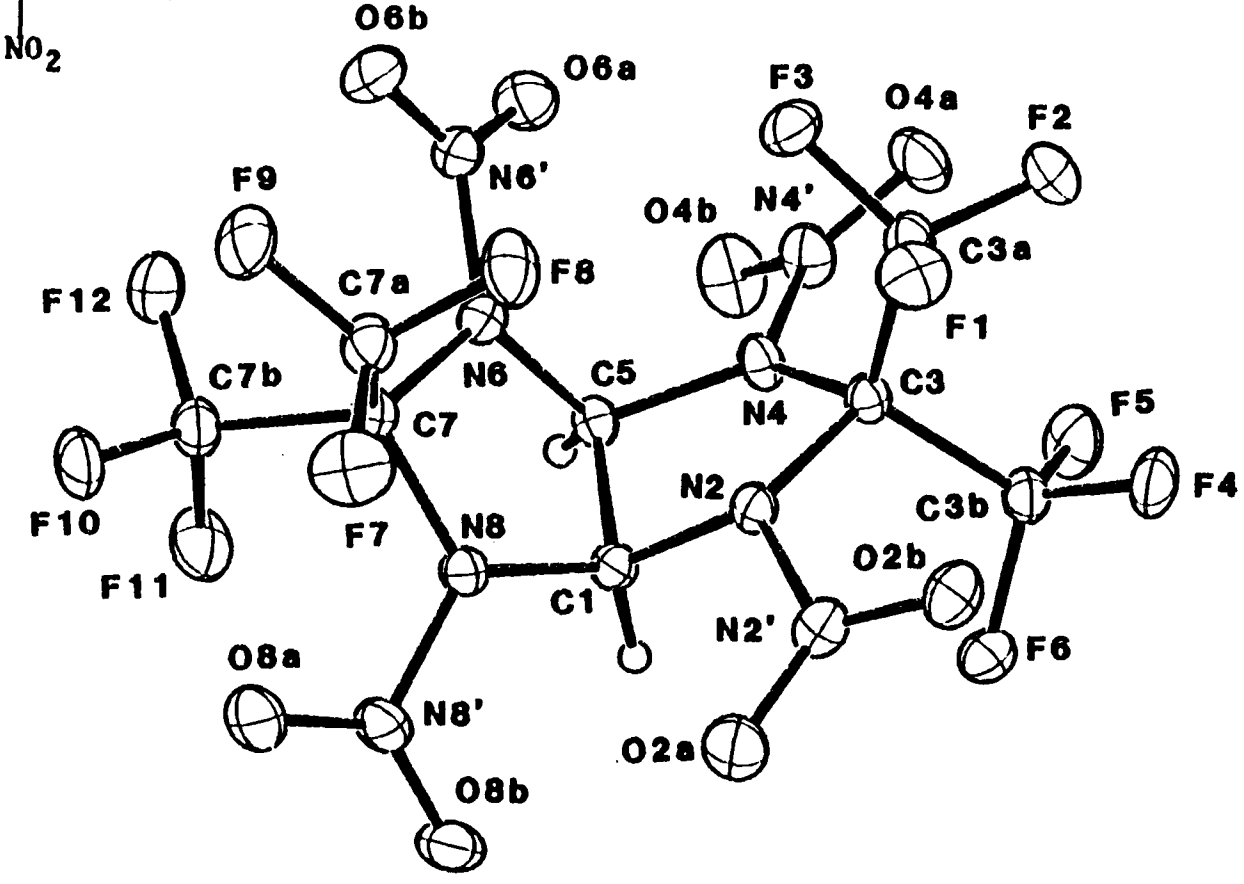
$C_8H_2N_8O_8F_{12}$, M_r = 566.13, monoclinic, C2/c, a = 34.074(5), b = 7.456(1), c = 13.877(2)Å, β = 102.41(1)°, m.p. = 110°C, V = 3443.1(8)Å^3, Z = 8, D_x = 2.184 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 2.413 mm^{-1}, F(000) =2224, T = 295K, Final R = 0.041, wR = 0.068 for 2799 independent observed reflections.

Experimental

Clear 0.50x0.35x0.12 mm. crystal, crystallized from CH_2Cl_2 /hexane, Synthesized by W. Koppes of Naval Surface Weapons Center (Silver Springs, Md.) Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 45<2θ<90 used for determining lattice parameters. Data corrected for Lorentz, polarization, and absorption effects, $(\sin\theta/\lambda)_{max}$ = 0.59Å^{-1}, range of hkl: -40<h<38, 0<k<8, 0<l<15. Standards 15 1 1,041,0 0 10, monitored every 60 reflections with linear variation 7.0% over data collection, θ-2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (5°/min. minimum, 30°/min. maximum), 3503 reflections measured, 2830 unique, R_{int} = 0.012, 2799 observed Fo>3σ(Fo). Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). Σ w(|Fo|-|Fc|)^2 minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00030, 333 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms, $(\Delta/\sigma)_{max}$ = 0.13, R = 0.041, wR = 0.068, S = 3.077. Final difference Fourier excursions 0.23 and -0.24eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



(E)



2,4,6,8-Tetranitro-3,3,7,7-tetra(trifluoromethyl)-

2,4,6,8-tetraazabicyclo(3.3.0)octane

3-Benzamido-6,8-dibenzoyl-1,3,5,6,8-pentaazabicyclo[3.2.2]nonane

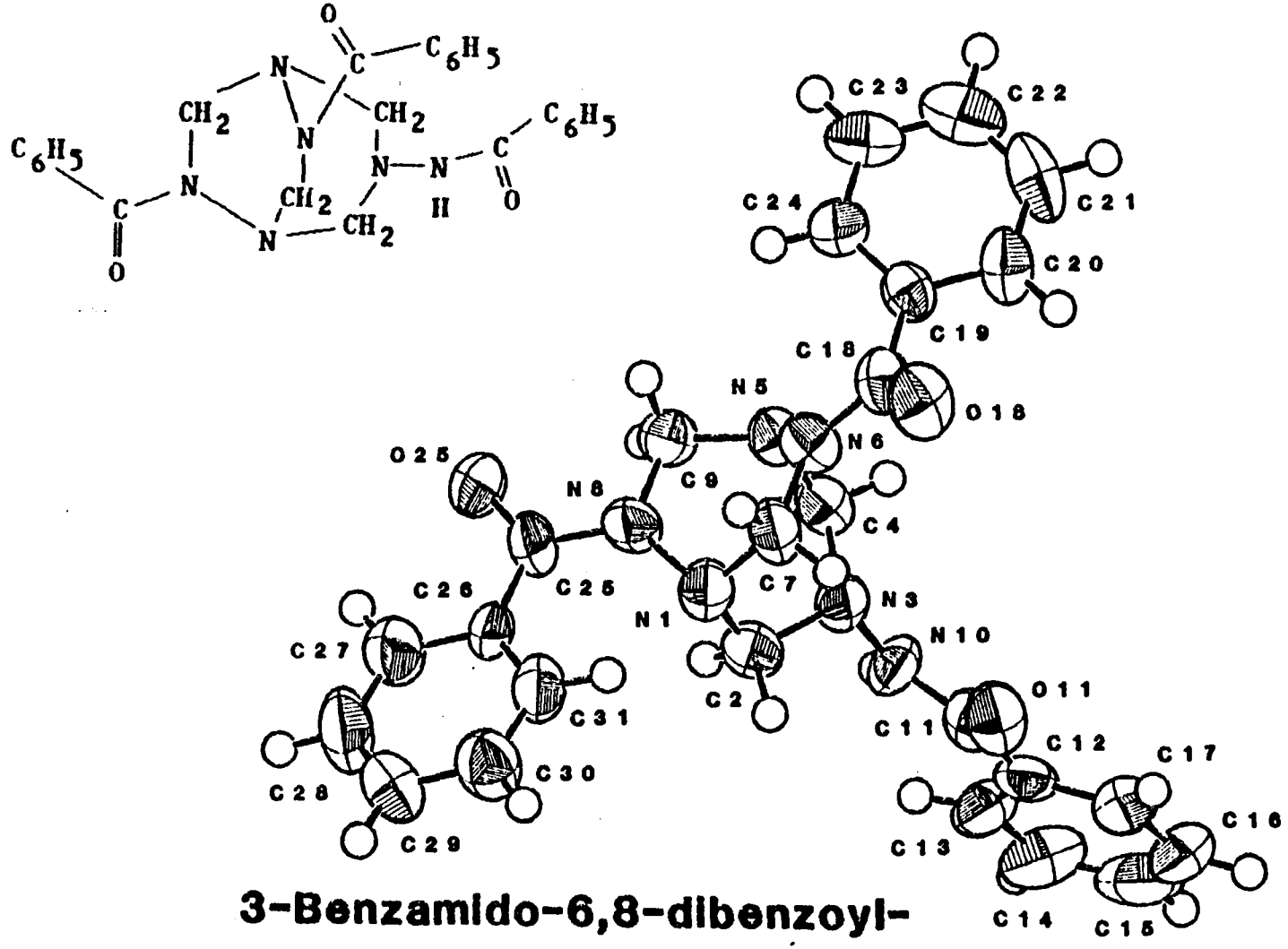
Abstract

$C_{25}H_{23}N_6O_3$, M_r = 455.50, monoclinic, $P2_1/c$, a = 11.325(3), b = 19.161(4), c = 11.952(5)Å, β = 112.01(3), V = 2280.3(10)Å^3. Z = 4, D_x = 1.327 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 0.70mm^{-1}, F(000) = 956, T = 295K°, Final R = 0.072, wR = 0.061 for 1269 independant observed reflections.

Experimental

Clear 0.05x0.02x0.15mm. crystal, crystallized from methanol. Synthesized by G. Kumar and J. Boyer of the University of Illinois at Chicago. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 20 centered reflections within 25<2θ<50 used for determining lattice parameters. Data corrected for Lorentz and polarization effects, $(\sin\theta/\lambda)_{max}$ = 0.53Å^{-1}, range of hkl: 0<h<10, 0<k<19, -12<l<11. Standards 202, 040, 002, monitored every 60 reflections with random variation 2.2% over data collection, θ-2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (2°/min. minimum. 30°/min. maximum), 2541 reflections measured, 2268 unique, R_{int} = 0.022, 1269 observed Fo>3σ(Fo). Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g*(Fo)^2]$, g = 0.00050 . 310 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, H atoms included using riding model, C-H = 0.96Å, H-C-H = 109.5°, U(H)= 1.2 U_{eq}(C), amine hydrogen coordinates, H(10),refined. $(\Delta/\sigma)_{max}$ = -0.19, R = 0.072, wR = 0.061, S = 1.293. Final difference Fourier excursions 0.24 and -0.22eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).

(F)



3-Benzamido-6,8-dibenzoyl-

1,3,5,6,8-pentaazabicyclo(3.2.2)nonane

1,3,5-Tribenzamido-1,3,5-hexahydrotriazine monohydrate

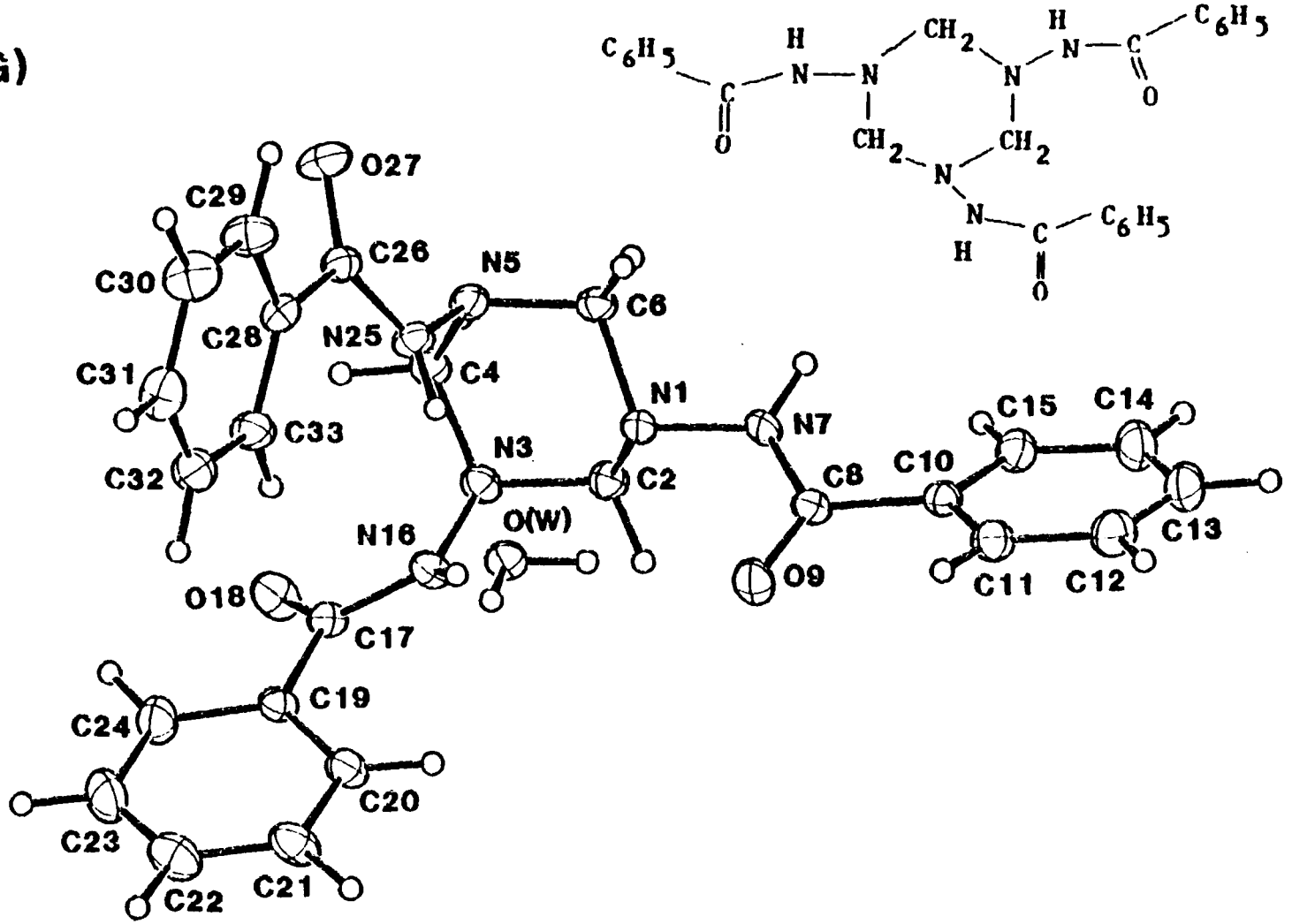
Abstract

$C_{24}H_{24}N_6O_3 \cdot H_2O$, M_r = 462.49, monoclinic, $P2_1/c$,
a = 12.655(1), b = 14.721(2), c = 13.339(2)Å, β = 107.25(1)°,
V = 2373.0(5)Å^3, Z = 4, D_x = 1.294 mg mm^{-3}, λ(CuKα) = 1.54178Å,
μ = 0.709 mm^{-1}, F(000) = 976, T = 295K, Final R = 0.042, wR = 0.045 for
3054 independent observed reflections.

Experimental

Clear 0.08x0.25x0.30 mm. crystal, crystallized from methanol/water. Synthesized by G.Kumar and J. Boyer of the Univ. of Ill. at Chicago. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178 Å(CuKα), 25 centered reflections within 37<2θ<90 used for determining lattice parameters. Data corrected for Lorentz and polarization, but not absorption effects, $(\sin\theta/\lambda)_{max}$ = 0.57Å^{-1}, range of hkl : -14<h<14, 0<k<16, -15<l<0. Standards $\bar{5}02$, $10\bar{6}$, 060, monitored every 60 reflections with random variation 1.3% over data collection, θ-2θ mode, scan width $(1.8 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (6°/min. minimum. 30°/min. maximum), 4156 reflections measured, 3799 unique, R_{int} = 0.011, 3054 observed Fo>3σ(Fo). Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00023, isotropic secondary extinction value, 0.00272, 412 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H. $(\Delta/\sigma)_{max}$ = -0.017, R = 0.042, wR = 0.045, S = 1.469. Final difference Fourier excursions 0.17 and -0.15eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).

(G)



1,3,5-Tribenzamido-1,3,5-hexahydrotriazine monohydrate

1-Benzoyl-4-benzamido-Δ^2-1,2,4-triazoline

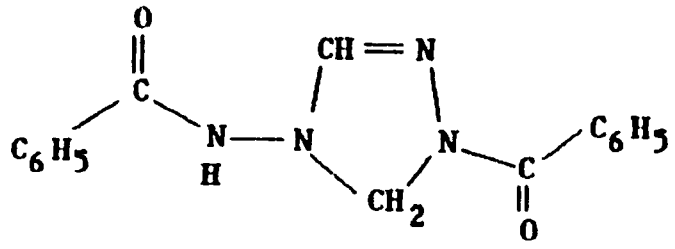
Abstract

$C_{15}H_{14}N_4O_2$, M_r = 294.31, monoclinic, $P2_1/c$, a =14.604(2), b = 10.339(1), c = 9.714(1)Å, β = 102.56(1)°, V = 1431.7(3)Å^3, Z = 4, D_x = 1.365 mg mm^{-3}, λ(Cu Kα) = 1.54178Å, μ = 0.73 mm^{-1}, F(000) = 616, T = 295K, Final R = 0.039, wR = 0.050 for 2117 independent observed reflections.

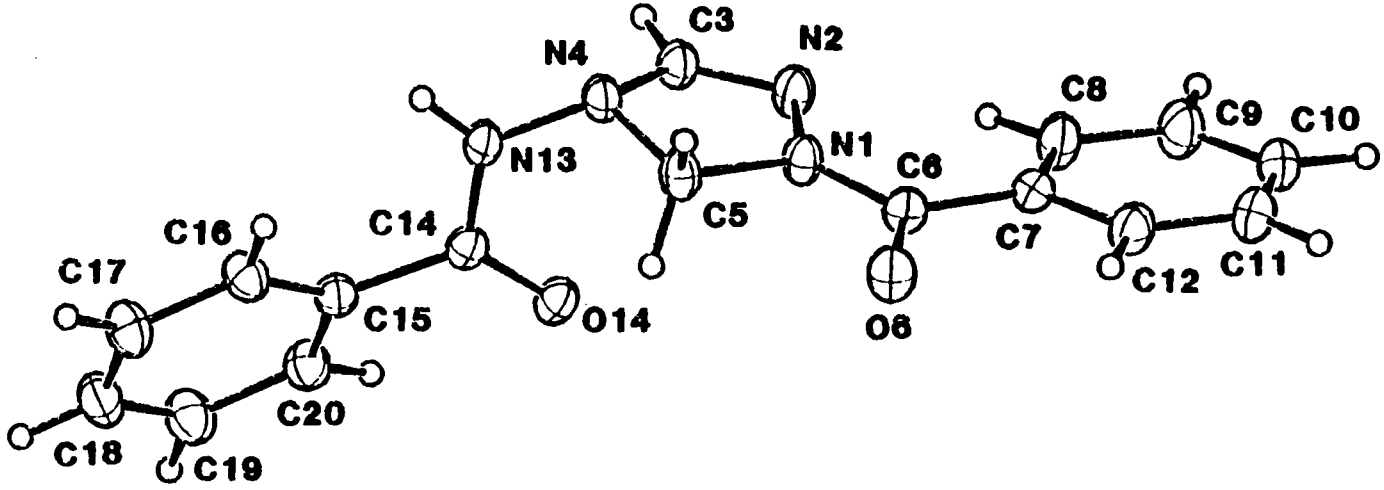
Experimental

Clear 0.15x0.25x0.40 mm. crystal, crystallized from benzene/chloroform. Synthesized by G. Kumar and J. Boyer of the Univ. of Ill. at Chicago, m.p. = 216-218 C°. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 30<2θ<60° used for determining lattice parameters. Data corrected for Lorentz and polarization effects. $(\sin\theta/\lambda)_{max}$ = 0.59Å^{-1}, range of hkl: -17<h< 1, 0<k<11, -11<l<11, Standards 800, 040, 004, monitored every 60 reflections with random variation 2.4% over data collection, θ-2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})$ scan rate a function of count rate (8°/min. minimum. 30°/min. maximum), 3408 reflections measured, 2437 unique, R_{int} = 0.014 , 2117 observed Fo>3σ(Fo).

Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). Σ w(|Fo|-|Fc|)^2 minimized where w = 1/[σ^2 (|Fo|) + g·(Fo)^2] , g = 0.0003, isotropic secondary extinction value = 0.00206 , 256 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H, (Δ/σ)max. = 0.016 ; R = 0.039, wR = 0.0503, S = 1.741 . Final difference Fourier excursions 0.19 and -0.16eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



(H)



1-Benzoyl-4-benzamido- Δ^2 -1,2,4-triazoline

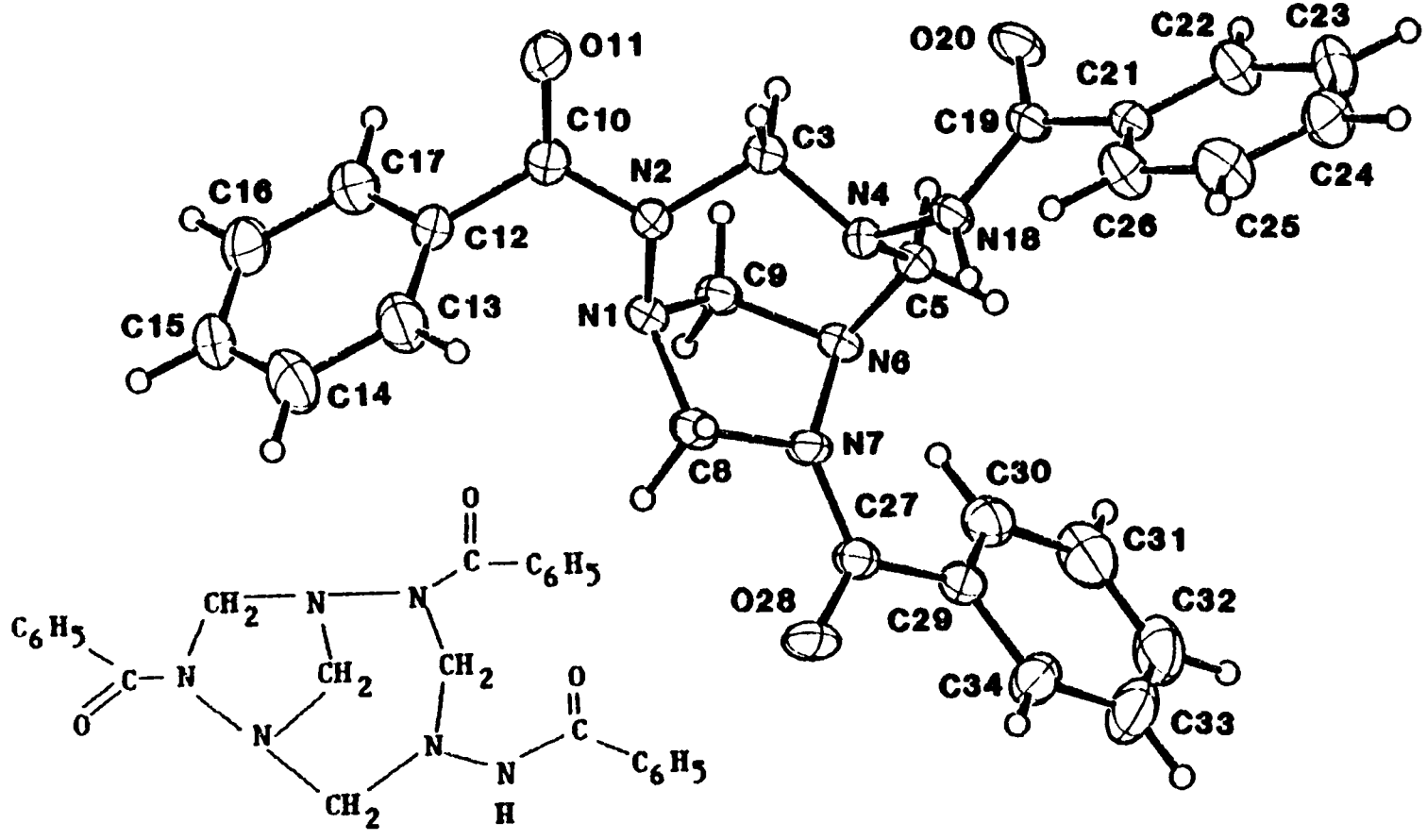
2,7-Dibenzoyl-4-benzamido-1,2,4,6,7-pentaazabicyclo[1.2.4]nonane

Abstract

$C_{25}H_{24}N_6O_3$, M_r = 456.51, triclinic, $P\bar{1}$, a = 9.195(1). b = 11.457(2), c = 12.139(2)Å, α = 89.77(2) , β = 72.79(1) , and γ = 76.52(1)°. V = 1184.9(4)Å^3, Z = 2, D_x = 1.279 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 0.674 mm^{-1}, F(000) = 480, T = 295K, Final R = 0.056, wR = 0.057 for 2421 independent observed reflections.

Experimental

Clear 0.32 x 0.21 x 0.08 mm. crystal, recrystallized from ethanol. Synthesized by G. Kumar and J. Boyer of the Univ. of Ill. at Chicago. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 39<2θ<60 used for determining lattice parameters. Data corrected for Lorentz, polarization, but not for absorption effects, $(\sin\theta/\lambda)_{max}$ = 0.57Å^{-1}, range of hkl: $-10<h<6$, $-12<k<11$, $-13<l<8$. Standards $00\bar{5}$, 060, $\bar{5}\bar{2}0$, monitored every 60 reflections with random variation 5.4% over data collection, θ-2θ mode, scan width $(1.8 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (8°/min. minimum, 30°/min. maximum), 3448 reflections measured, 3131 unique, R_{int} = 0.018, 2421 observed Fo>3σ(Fo). Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00023 , isotropic secondary extinction value, 0.0078 , 344 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H , benzene ring hydrogens included using riding model, C-H = 0.96Å, C-C-H = 120.0°, 1.1 U_{eq}(C). $(\Delta/\sigma)_{max}$ = -0.008, R = 0.056, wR = 0.057, S = 1.512. Final difference Fourier excursions 0.40 and -0.23 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974



(I)

2,7-Dibenzoyl-4-benzamido-

1,2,4,6,7-pentaazabicyclo(1.2.4)nonane

bis-Tetrazolo[1,5-a:1',5'-c]pyrazine

Abstract

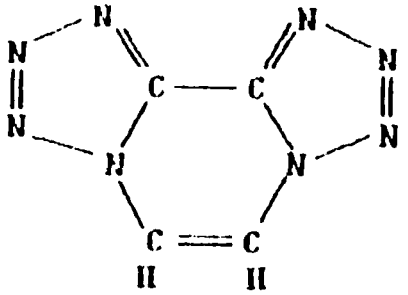
$C_4H_2N_8$, M_r = 162.11, orthorhombic, $P2_12_12_1$, a = 6.1155(5), b = 8.2572(8), c = 12.7401(11)Å, V = 643.3(1)Å^3, Z = 4, D_x = 1.674 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 1.036 mm^{-1}, F(000) = 328, T = 295K° Final R = 0.0326, wR = 0.0440 for 1106 independent observed reflections. All of the non-hydrogen atoms in this new heteroaromatic compound lie within 0.014Å of a common plane.

Experimental

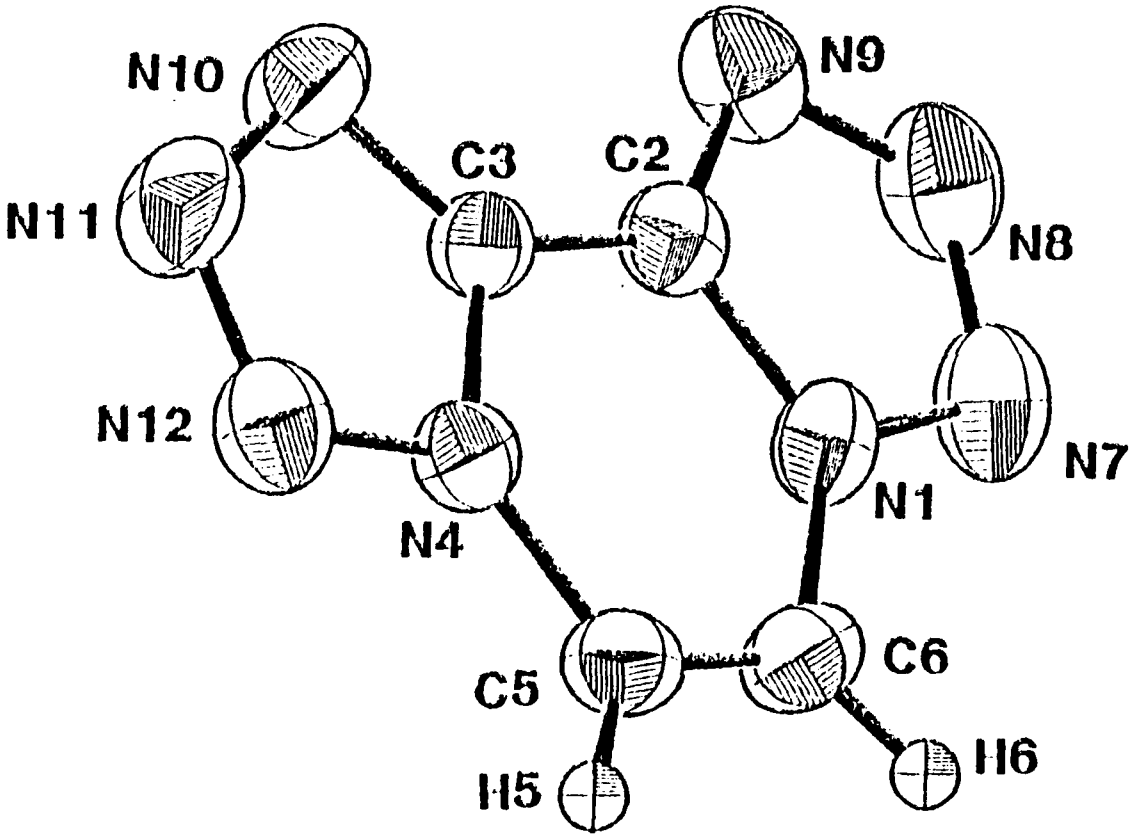
Clear, colorless square prisms; data crystal 0.15 x 0.15 x 0.55mm., synthesized & crystallized by M. Chaykovsky of Naval Surface Weapons Center (Silver Spring, Md.). Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα); 25 centered reflections within 43<2θ<75° used for determining lattice parameters. Data corrected for Lorentz & polarization, but not absorption effects, $(2\theta)_{max}$ = 139°, range of hkl: 0<h<7, -9<k<10, -15<l<15. Standards 400, 040, 0 0 10, monitored every 60 reflections with random variation ±4% over data collection, θ/2θ mode, scan width $(1.4 + \Delta_{\alpha 1 \alpha 2})°$, scan rate a function of count rate (4°/min. minimum, 30°/min. maximum), 1976 reflections measured (incl. 97 monitors), 1106 unique (Friedel pairs not merged), R_{int} = 0.0178, no reflections considered unobserved.

Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.000225, isotropic secondary extinction correction applied, 116 parameters refined: atom coordinates (all atoms),

anisotropic temperature factors for all non-H atoms, H atom U_{ij}'s fixed at anisotropic values predicted by rigid-body vibration analysis of non-H atoms, (Δ/σ) max = 0.005, R = 0.0326, wR = 0.0440, S = 2.25. Final difference Fourier extrema 0.38 and -0.28 eA^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



(J)



bis-Tetrazolo(1,5-a:1'5'-c)pyrazine

1,4-Dinitro-2,3,5,6-tetrabromo-2,3,5,6-piperazine

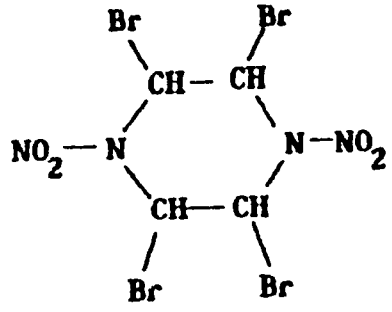
Abstract: $C_6H_6N_4O_4Br_4$, M_r = 491.72, triclinic, $P\bar{1}$, a = 7.517(3), b = 7.566(2), c = 11.835(3) Å, α = 78.89(2), β = 77.44(3), γ = 63.84(2)°, F(000) = 456, V = 586.1(3) $Å^3$, z = 2, D_x = 2.79 mg mm^{-3}, λ (CuKα) = 1.54178 Å, μ = 170.96 cm^{-1}, T = 295K, final R = 0.069 for 1891 unique reflections. Asymmetric unit consisted of two half-molecules. Results showed the bromine atoms are in trans-trans configuration, and are all axial with respect to the piperazone chair-shaped ring.

Experimental: Clear colorless 0.23x0.15x0.08 mm crystal provided by M. Chaykovsky of the Naval Surface Weapons Center (Silver Spring, MD). Nicolet R3M diffractometer, monochromator on incident beam, θ-2θ scan technique with a constant scan speed of 60°/min. Unit cell parameters from least-squares analysis of 25 reflections with 2θ from 47 to 95°. $P\bar{1}$ checked for higher symmetry using program AIDS (Mighell, Hubbard and Stalick, 1980), 2038 independent reflections with $2\theta_{max}$ = 130°; three standard reflections measured after every 60 new reflections showed an average random variation of 3.0% in $|F_o|$. Dm not determined, crystals sank in CCl_4 (d = 1.59 mg mm^{-3}), Lorentz polarization corrections, empirical absorption correction applied (max. trans = 0.913, min trans = 0.363). Structure solved by direct methods (Karle and Karle, 1966), Br atoms found in E-map, all remaining non-hydrogen atoms in a difference map. Refined by full-matrix least-squares, function minimized $\Sigma w(|F_o|-|F_c|)^2$, isotropic secondary extinction correction applied. Non-H atoms anisctropic, H atoms in calculated positions riding on bonded atoms, 1891 reflections having $|F_o|>3\sigma|F_o|$ refined to a final R factor of 0.069 (Rw = 0.083, S = 3.0), Δ/σ_{max} = 0.20, final difference map

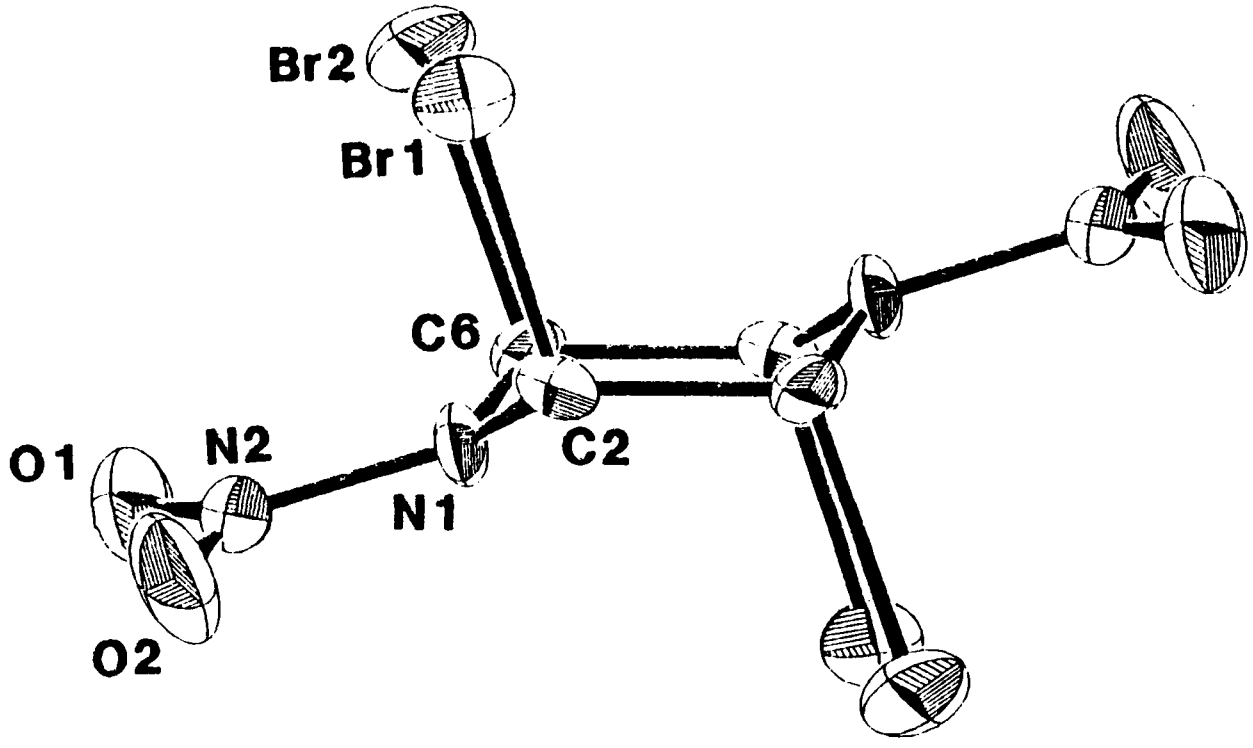
was featureless except for ripples around Br atoms (max height = 1.76 A^{-3}).

Discussion

The asymmetric unit for this compound consisted of 2 half-molecules located on centers of symmetry rather than one full molecule in a general position. However, there are no significant differences in the conformations of the two molecules. In both molecules the six-membered ring has a slightly flattened chair conformation and the NO_2 group is coplanar with N1. The sum of angles around both N2 and N2' is 359.9. Pairs of bromine atoms substituted on adjacent carbon atoms of the ring are <u>trans</u> with respect to one another, and are axial with respect to the ring. Amino nitrogen atoms substituted with nitro groups are usually near planar in geometry. In this molecule, the amino nitrogens are slightly pyramidal, and the nitro groups both bend in the equatorial direction with respect to the ring. Even though this crystal has a very high density (d_{calc} = 2.79) there are only a few close intermolecular contacts: a Br1···Br2 approach at 3.69 Å and a Br2···O2' approach at 3.28 Å.



(K)



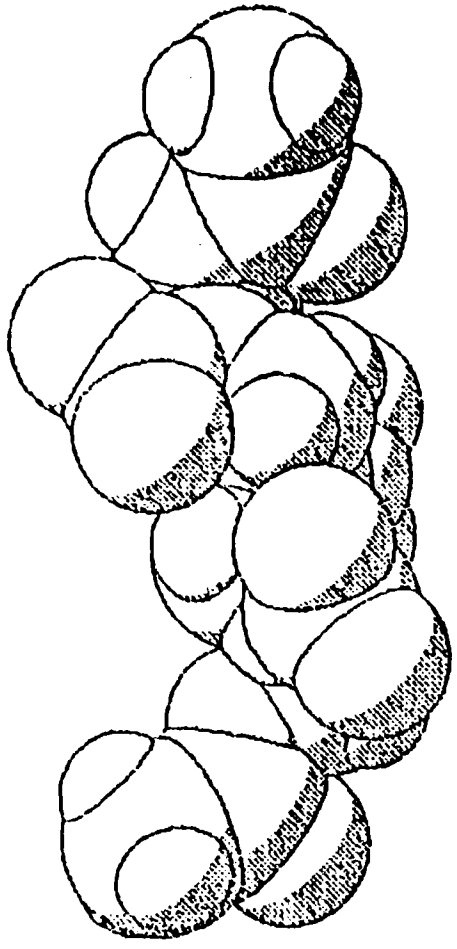
1,4-Dinitro-2,3,5,6-tetrabromopiperazine

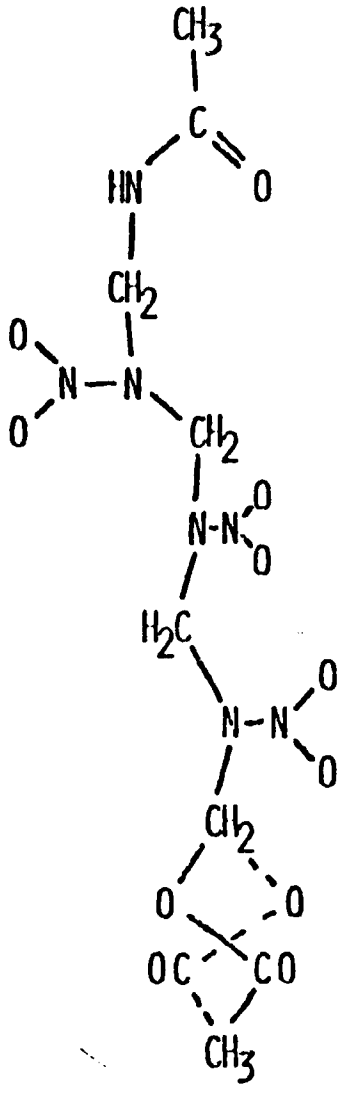
1,9-Diacetyl-3,5,7-trinitro-1,3,5,7-tetraaza-9-oxanonane

Abstract

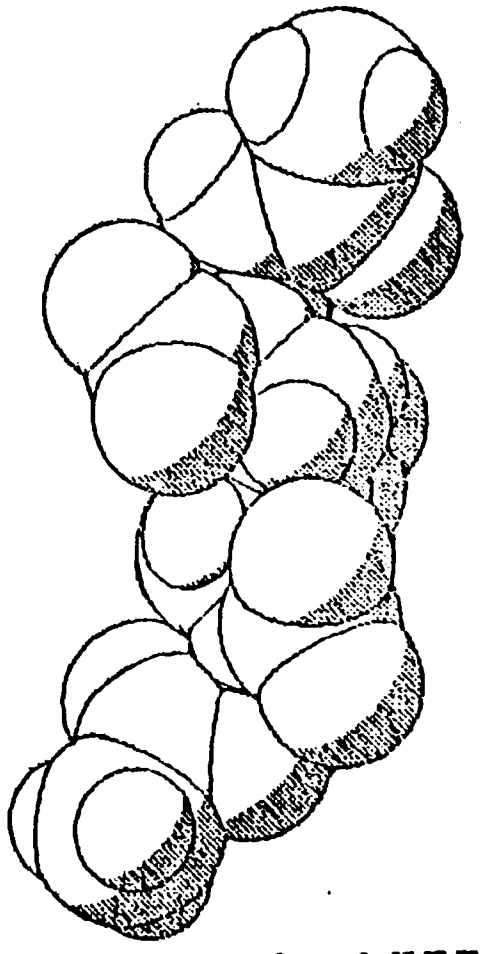
$C_8H_{15}N_7O_9$, M_r = 353.25, orthorhombic Pbca, a = 12.410(2), b = 9.695(1), c = 25.079(3)Å, α = β = γ = 90.00°, V = 3017.5(6)Å^3, Z = 8, D_x = 1.555 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 1.189 mm^{-1}, F(000) = 1472, T = 295K, Final R = 0.087, wR = 0.094 for 1427 independent observed reflections. The acetoxy group is disordered with an occupancy of 57 and 43% respectively for the two orientations.

Clear 0.05 x 0.10 x 0.25 mm. crystal crystallized from methyl chloride. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 30<2θ<50° used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.54Å^{-1}, range of hkl: -13<h<1, 0<k<10, 0<l<14. Standards 400, 040, 006, monitored every 60 reflections with random variation 4.6% over data collection, θ/2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (2°/min. minimum, 30°/min. maximum), 2782 reflections measured, 2082 unique, R_{int} = 0.02, 1427 observed Fo>3σ(Fo). Data corrected for Lorentz, polarization but not absorption effects, Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00040, Secondary extinction value, 0.0009. 356 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, H atoms included using riding model, C-H = 0.96Å, H-C-H = 109.5°, U(H)= 1.2 U_{eq}(C), $|(\Delta/\sigma)_{max}$ = 0.123, R = 0.066, wR = 0.068, S = 1.841. Final difference Fourier excursions 0.38 and -0.34 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).





(L)



d - 1.555

1,9-DIACETYL-3,5,7-TRINITRO-1,3,5,7-TETRAAZA-9-OXANONANE

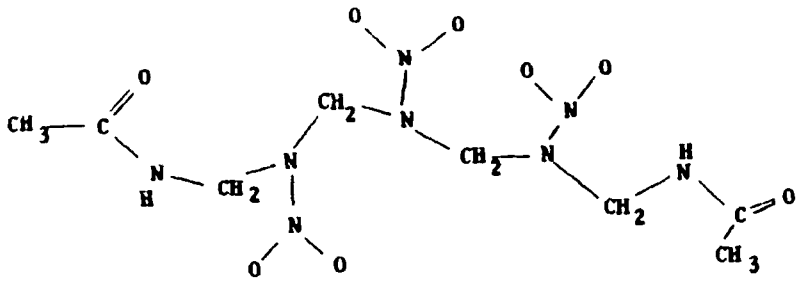
1,9-Diacetyl-3,5,7-trinitro-1,3,5,7,9-pentaazanonane

Abstract

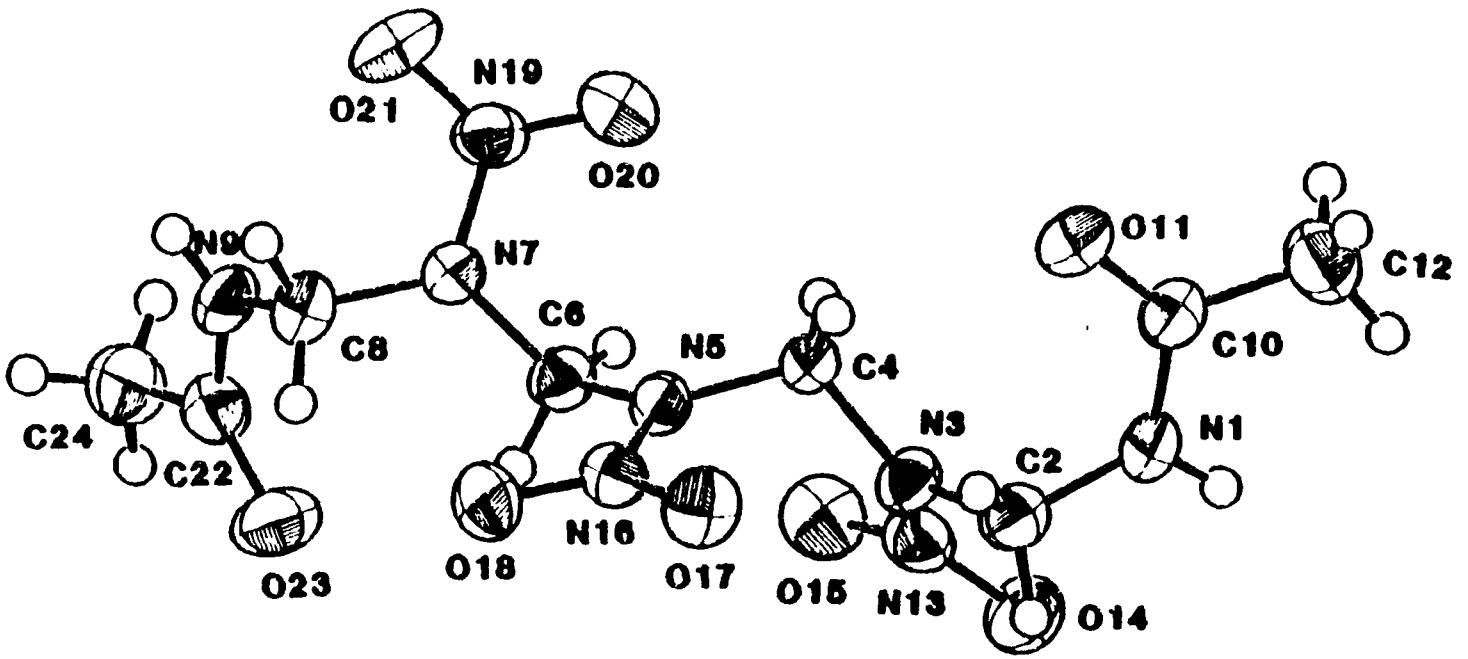
$C_8H_{16}N_8O_8$, M_r = 352.17, monoclinic, $P2_1$, a = 7.243(2), b = 19.986(4), c = 10.692(2)Å, β = 96.18(2)°, V = 1538.7(6)Å^3, Z = 4, D_x = 1.520 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 1.13 mm^{-1}, F(000) = 736, T = 295K. Final R = 0.038 wR = 0.045 for 2900 independent observed reflections. There are two independent molecules in the asymmetric unit.

Clear 0.25 x 0.15 x 0.45 mm. crystal crystallized from methyl chloride. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 40<2θ<65° used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.61Å^{-1}, range of hkl:-7<h<8 ,-24<k<0, -12<l<1 . Standards $\bar{2}0\bar{3}$, $0\bar{8}0$, $00\bar{5}$, monitored every 60 reflections with random variation 1.4% over data collection, θ/2θ mode. scan width $(2.0 + \Delta_{\alpha 1 \alpha 2})$°, scan rate a function of count rate (4°/min. minimum, 30°/min. maximum), 4272 reflections measured, 2982 unique, R_{int} = 0.027, 2900 observed Fo>3σ(Fo). Data corrected for Lorentz, polarization and absorption effects, Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). Σ w(|Fo|-|Fc|)^2 minimized where w = 1/[σ^2 (|Fo|) + g·(Fo)^2] , g = 0.00030.

458 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, amine hydrogen coordinates and isotropic temperature factors, other H atoms included using riding model, C-H = 0.96Å, angle H-C-H = 109.5°, U(H)= 1.2 U_{eq}(C).(Δ/σ) max = 0.40, mean = 0.03, R = 0.038, wR = 0.045, S = 1.737. Final difference Fourier excursions 0.14 and -0.31eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



(M)



d - 1.52

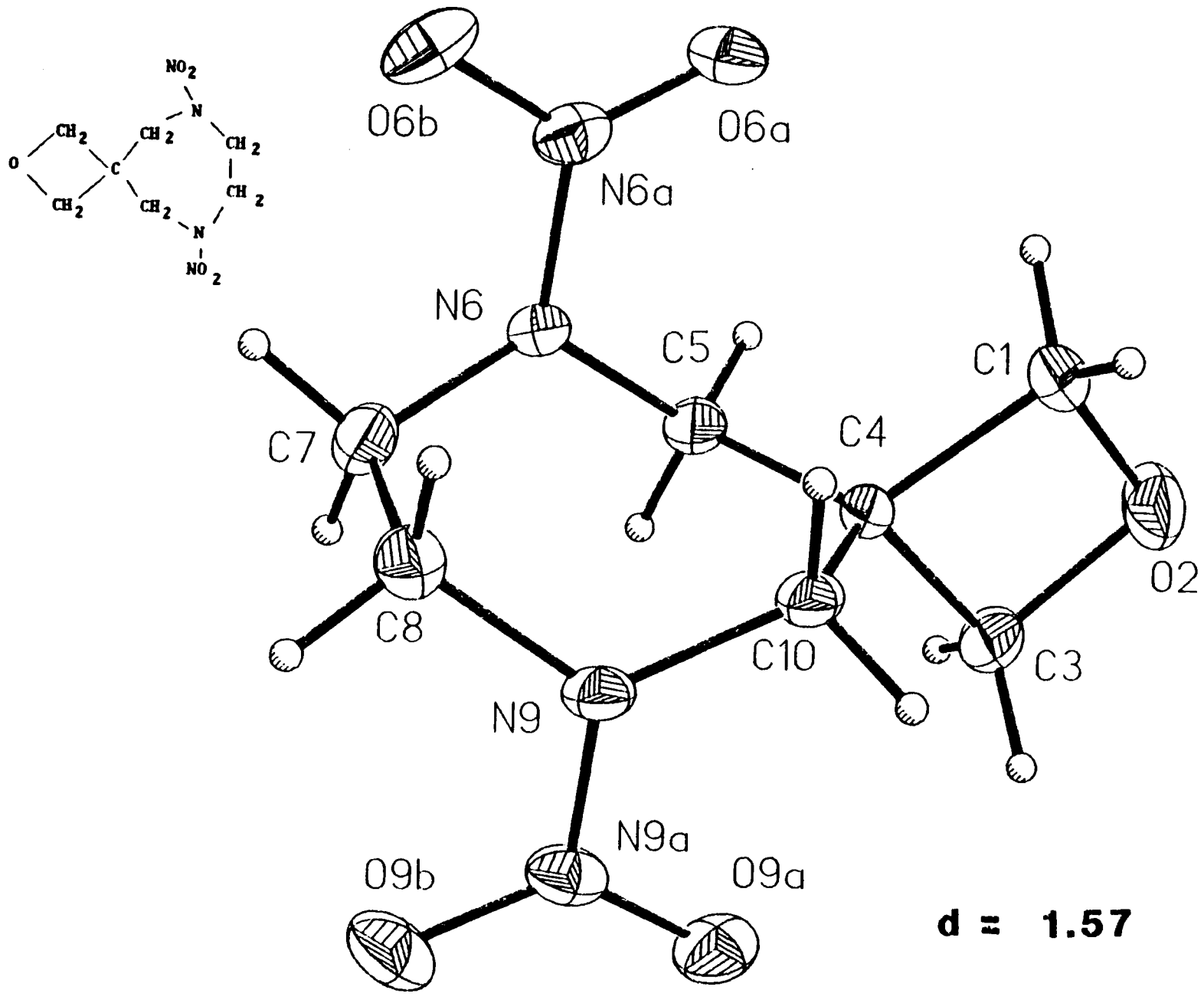
1,9-Diacetyl-3,5,7-trinitro-1,3,5,7,9-pentaazanonane

2-Oxa-6,9-diaza-6,9-dinitrospiro[3.6]decane

Abstract

$C_7H_{12}N_4O_5$, M_r = 232.20, triclinic, $P\bar{1}$, a = 6.028(1), b = 6.420(2), c = 13.764(4)Å, α = 91.04(2), β = 100.22(2), and γ = 108.57(2)°, V = 492.0(2)Å^3, Z = 2, D_x = 1.567 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 1.108 mm^{-1}, F(000) = 244, T = 295K, Final R = 0.075, wR = 0.075 for 877 independent observed reflections.

Clear 0.02 x 0.08 x 0.15 mm. crystal crystallized from ethyl acetate, Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 32<2θ<75° used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.58Å^{-1}, range of hkl: 0<h<6 ,-7<k<7, -15<l<15. Standards $0\bar{2}1$, 200, 003. monitored every 60 reflections with random variation 2.5% over data collection, θ/2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (3°/min. minimum, 30°/min. maximum), 1864 reflections measured, 1253 unique, R_{int} = 0.026, 877 observed Fo>3σ(Fo). Data corrected for Lorentz, polarization and absorption effects, Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). Σ w(|Fo|-|Fc|)^2 minimized where w = 1/[σ^2 (|Fo|) + g·(Fo)^2] , g = 0.00063 , Secondary extinction value 0.0022. 146 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms isotropic temperature factors for H atoms [H atoms included using riding model, C-H = 0.96Å, H-C-H = 109.5°, U(H)= 1.1 U_{eq}(C)], (Δ/σ) max = 0.01 , R = 0.075, wR = 0.075, S = 1.64 . Final difference Fourier excursions 0.28 and -0.29eÅ-3 . Atomic scattering factors from International Tables for X-ray Crystallography (1974).



2-Oxa-6,9-diaza-6,9-dinitrospiro(3.6)decane

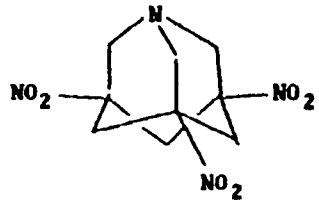
1-Aza-3,5,7-trinitroadamantane

Abstract

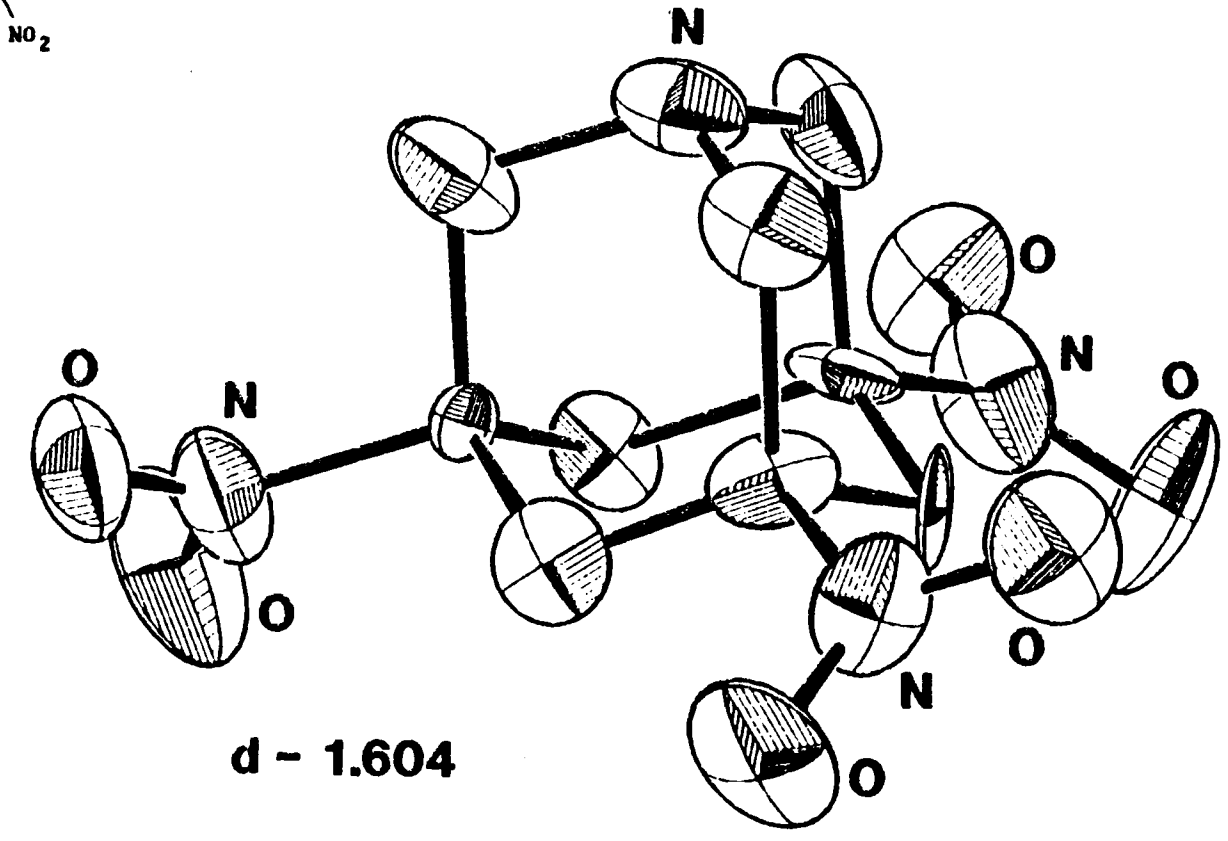
$C_9H_{12}N_4O_6$, M_r = 272.22, trigonal, a = b = 12.821(3), c = 5.937Å, α = 90.00, β = 90.00, and γ = 120.00°, V = 845.2(4)Å^3, Z = 3, D_x = 1.604 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 1.13 mm^{-1}, F(000) = 426, T = 295K. The cage nitrogen lies on a three fold axis.

Clear 0.15 x 0.08 x 0.20 mm. crystal. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα, 20 centered reflections within 20<2θ<50° used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.59 Å^{-1}, range of hkl:-15<h<12, -14<k<12, -6<l<0. Standards $C0\bar{3}$, $3\bar{2}0$, $\bar{1}30$, monitored every 60 reflections with random variation 3.5% over data collection, θ/2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (2°/min. minimum, 30°/min. maximum), 3016 reflections measured, 1017 unique, R_{int} = 0.035 . Data corrected for Lorentz, polarization but not absorption effects, Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g*(Fo)^2]$, g = 0.00060.

The structure analysis is not yet complete, however, atomic assignment is definitive.



(O)



d - 1.604

1-Aza-3,5,7-trinitroadamantane

7-Nitro-5,6-dihydro-7H-imidazolo[1,2-d]tetrazole

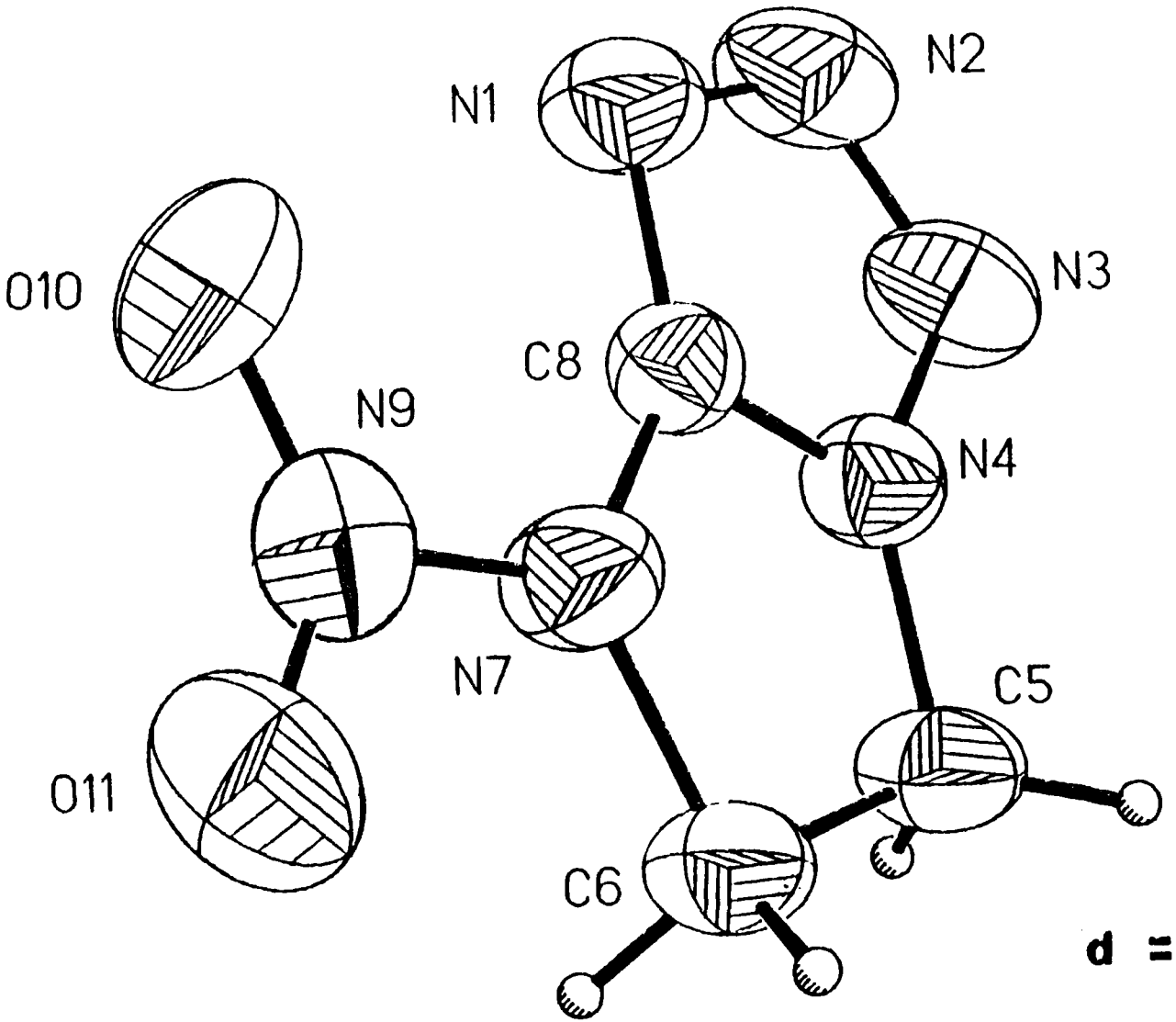
Abstract

$C_3H_4N_6O_2$, M_r = 156.11, monoclinic, $P2_1/c$, a = 6.214(2), b = 8.692(3), c = 11.664(3)Å, β = 102.42(2)°. Decomposes without melting. V = 615.2(3)Å^3, Z = 4, D_x = 1.685 mg mm^{-3}, λ(CuKα) = 1.54178Å, μ = 1.195 mm^{-1}, F(000) = 320, T = 295K, Final R = 0.041, wR = 0.060 for 986 independent observed reflections. The compound has a whole molecule disorder with a 12% occupancy. The disordered molecule is related to the primary molecule via a pseudo two fold axis passing midway through the N(7)- C(8) and C(5)-C(6) bonds.

Experimental

Clear 0.60 x 0.12 x 0.08 mm. crystal crystallized from acetone. Automated Nicolet R3m diffractometer with incident beam graphite monochromator λ = 1.54178Å(CuKα), 25 centered reflections within 30<2θ<73 used for determining lattice parameters. Data corrected for Lorentz and polarization effects, $(\sin\theta/\lambda)_{max}$ = 0.59Å^{-1}, range of hkl: -7<h<0, 0<k<9, -13<l<13. Standards $\bar{3}00$, 040, 008, monitored every 60 reflections with random variation 2.6% over data collection, θ/2θ mode, scan width $(2.0 + \Delta_{\alpha1\alpha2})°$, scan rate a function of count rate (4°/min. minimum, 30°/min. maximum), 1243 reflections measured, 1049 unique, R_{int} = 0.011, 986 observed Fo>3σ(Fo). Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). Σ w(|Fo|-|Fc|)2 minimized where $w = 1/[\sigma^2(|Fo|) + g\cdot(Fo)^2]$, g = 0.00023, 130 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms. (Δ/σ) max = 0.10, R = 0.041, wR = 0.060, S = 2.657. Final difference Fourier excursions 0.13 and -0.14 eÅ^{-3} . Atomic scattering factors from International Tables for X-ray Crystallography (1974).

(P)



d = 1.69

7-Nitro-5,6-dihydro-7H-Imidazolo(1,2-d)tetrazole

Appendix A

This appendix list atom coordinates in a fractional or 'crystallograpic' coordinate system. In addition tables of bond distances and angles as well as tables of anisotropic thermal parameters are provided. The alphabetic labels follow those given in the index.

TABLE 1a Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
N(1)	2878(6)	6381(6)	5825(10)	43(2)*
C(2)	2750(7)	6067(7)	7668(12)	38(3)*
N(3)	1436(6)	5548(8)	7676(10)	42(2)*
C(4)	837(7)	5543(7)	6118(12)	36(3)*
C(5)	1735(8)	6080(8)	4754(13)	44(3)*
N(6)	3908(7)	7050(7)	5336(12)	53(3)*
O(7)	3875(6)	7334(7)	3769(11)	66(3)*
C(8)	2802(8)	7150(9)	8882(14)	55(3)*
C(9)	3721(8)	5036(8)	8144(15)	49(3)*
O(10)	-244(5)	5108(6)	5841(8)	49(2)*
F(11)	2358(6)	6872(6)	10454(8)	81(2)*
F(12)	3998(5)	7593(5)	9110(10)	79(2)*
F(13)	2091(5)	8060(5)	8259(11)	85(2)*
F(14)	4933(5)	5409(5)	8169(10)	76(2)*
F(15)	3600(6)	4122(5)	7057(10)	83(3)*
F(16)	3442(5)	4610(6)	9745(10)	81(2)*
Hn(3)	1025(83)	5432(82)	8627(166)	61(29)

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2a Bond lengths (A)

N(1)-C(2)	1.451(11)	N(1)-C(5)	1.461(11)
N(1)-N(6)	1.337(10)	C(2)-N(3)	1.457(10)
C(2)-C(8)	1.508(13)	C(2)-C(9)	1.549(12)
N(3)-C(4)	1.335(11)	N(3)-Hn(3)	0.847(116)
C(4)-C(5)	1.507(12)	C(4)-O(10)	1.221(9)
N(6)-O(7)	1.233(12)	C(8)-F(11)	1.315(12)
C(8)-F(12)	1.326(10)	C(8)-F(13)	1.324(11)
C(9)-F(14)	1.304(10)	C(9)-F(15)	1.307(12)
C(9)-F(16)	1.336(13)		

TABLE 3a Bond angles (deg.)

C(2)-N(1)-C(5)	114.4(6)	C(2)-N(1)-N(6)	118.1(7)
C(5)-N(1)-N(6)	126.7(7)	N(1)-C(2)-N(3)	100.4(6)
N(1)-C(2)-C(8)	113.7(7)	N(3)-C(2)-C(8)	109.8(7)
N(1)-C(2)-C(9)	110.1(7)	N(3)-C(2)-C(9)	107.6(7)
C(8)-C(2)-C(9)	114.3(7)	C(2)-N(3)-C(4)	114.8(7)
C(2)-N(3)-Hn(3)	121.3(69)	C(4)-N(3)-Hn(3)	122.1(69)
N(3)-C(4)-C(5)	109.4(7)	N(3)-C(4)-O(10)	124.7(8)
C(5)-C(4)-O(10)	125.8(8)	N(1)-C(5)-C(4)	101.0(7)
N(1)-N(6)-O(7)	112.8(7)	C(2)-C(8)-F(11)	111.2(8)
C(2)-C(8)-F(12)	113.7(7)	F(11)-C(8)-F(12)	106.5(8)
C(2)-C(8)-F(13)	110.9(8)	F(11)-C(8)-F(13)	108.2(8)
F(12)-C(8)-F(13)	106.0(7)	C(2)-C(9)-F(14)	112.4(7)
C(2)-C(9)-F(15)	110.7(8)	F(14)-C(9)-F(15)	109.9(8)
C(2)-C(9)-F(16)	109.5(7)	F(14)-C(9)-F(16)	107.4(8)
F(15)-C(9)-F(16)	106.8(7)		

TABLE 4a Anisotropic temperature factors ($A^2 \times 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	31(3)	65(5)	32(4)	6(4)	2(3)	-17(3)
C(2)	26(4)	52(5)	36(5)	2(4)	-1(4)	1(4)
N(3)	31(4)	73(5)	23(4)	-3(4)	-7(3)	-12(4)
C(4)	34(4)	39(5)	34(5)	-3(4)	-2(3)	-1(4)
C(5)	37(4)	66(6)	28(4)	-9(5)	3(4)	-1(4)
N(6)	44(4)	69(5)	45(5)	10(4)	0(4)	-6(4)
O(7)	61(4)	89(5)	49(5)	12(4)	10(4)	-12(4)
C(8)	50(5)	55(6)	59(6)	-2(5)	-2(5)	-10(5)
C(9)	44(5)	51(5)	51(6)	-3(5)	-15(5)	-4(4)
O(10)	34(3)	78(4)	34(3)	-3(3)	-9(3)	-7(3)
F(11)	99(4)	112(5)	33(3)	-21(3)	20(3)	-26(4)
F(12)	59(3)	90(4)	87(4)	-28(3)	-7(3)	-31(3)
F(13)	98(4)	70(4)	88(4)	-13(4)	-8(4)	29(3)
F(14)	32(2)	89(4)	108(5)	23(4)	-12(3)	-8(3)
F(15)	88(4)	65(3)	96(5)	-16(4)	-18(4)	8(3)
F(16)	69(4)	109(4)	65(4)	38(4)	-4(3)	10(3)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$$

TABLE 5a Hydrogen coordinates ($\times 10^4$) and temperature factors ($A^2 \times 10^3$)

atom	x/a	y/b	z/c	U
H(5a)	1362	6793	4224	53
H(5b)	1947	5499	3857	53

TABLE 1b Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
C(1x)	7105(3)	505(4)	7247(1)	44(1)*
N(2x)	5985(2)	1662(4)	7365(1)	48(1)*
C(3x)	5844(3)	1846(4)	7922(1)	42(1)*
N(4x)	7099(2)	1412(4)	8137(1)	52(1)*
C(3ax)	5532(3)	3917(5)	8044(1)	51(1)*
C(3bx)	4811(3)	518(5)	8128(1)	57(1)*
F(1x)	4429(2)	4464(3)	7839(1)	72(1)*
F(2x)	5481(2)	4252(3)	8550(1)	73(1)*
F(3x)	6436(2)	5065(3)	7865(1)	83(1)*
F(4x)	3682(2)	785(3)	7901(1)	80(1)*
F(5x)	4642(2)	690(4)	8633(1)	92(1)*
F(6x)	5134(2)	-1267(3)	8042(1)	97(1)*
C(1)	9188(3)	5485(4)	9432(1)	40(1)*
N(2)	10493(2)	6151(3)	9293(1)	40(1)*
C(3)	10620(3)	5871(4)	8738(1)	42(1)*
N(4)	9338(2)	5693(4)	8520(1)	49(1)*
C(5)	8412(3)	5586(4)	8929(1)	42(1)*
N(6)	7662(2)	7331(3)	8997(1)	44(1)*
C(7)	7545(3)	7738(4)	9544(1)	42(1)*
N(8)	8602(2)	6751(3)	9795(1)	44(1)*
C(3a)	11310(4)	7537(5)	8492(1)	67(1)*
C(3b)	11386(3)	4033(5)	8663(1)	56(1)*
C(7a)	7727(4)	9856(5)	9636(1)	59(1)*
C(7b)	6260(3)	7044(5)	9757(1)	57(1)*
F(1)	12385(3)	7972(4)	8721(1)	126(1)*
F(2)	11514(2)	7297(3)	7998(1)	97(1)*
F(3)	10568(3)	9089(3)	8526(1)	109(1)*
F(4)	12586(2)	4190(4)	8830(1)	97(1)*
F(5)	11440(2)	3496(3)	8167(1)	69(1)*
F(6)	10856(2)	2640(3)	8922(1)	95(1)*
F(7)	7720(2)	10333(3)	10131(1)	86(1)*
F(8)	8835(2)	10437(3)	9450(1)	94(1)*
F(9)	6817(2)	10886(3)	9397(1)	89(1)*
F(10)	6125(2)	7377(3)	10257(1)	82(1)*
F(11)	6171(2)	5173(3)	9692(1)	85(1)*
F(12)	5266(2)	7801(4)	9514(1)	96(1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2b Bond lengths (A)

C(1x)-N(2x)	1.452(4)	C(1x)-C(1xa)	1.531(6)
C(1x)-N(4xa)	1.460(4)	N(2x)-C(3x)	1.447(4)
C(3x)-N(4x)	1.452(4)	C(3x)-C(3ax)	1.532(5)
C(3x)-C(3bx)	1.528(5)	N(4x)-C(1xa)	1.460(4)
C(3ax)-F(1x)	1.325(4)	C(3ax)-F(2x)	1.317(4)
C(3ax)-F(3x)	1.329(4)	C(3bx)-F(4x)	1.321(4)
C(3bx)-F(5x)	1.323(4)	C(3bx)-F(6x)	1.321(4)
C(1)-N(2)	1.435(4)	C(1)-C(5)	1.524(4)
C(1)-N(8)	1.487(4)	N(2)-C(3)	1.447(4)
C(3)-N(4)	1.446(4)	C(3)-C(3a)	1.524(5)
C(3)-C(3b)	1.537(4)	N(4)-C(5)	1.470(4)
C(5)-N(6)	1.443(4)	N(6)-C(7)	1.448(4)
C(7)-N(8)	1.451(4)	C(7)-C(7a)	1.523(5)
C(7)-C(7b)	1.535(5)	C(3a)-F(1)	1.321(4)
C(3a)-F(2)	1.340(4)	C(3a)-F(3)	1.321(4)
C(3b)-F(4)	1.316(4)	C(3b)-F(5)	1.319(4)
C(3b)-F(6)	1.334(4)	C(7a)-F(7)	1.299(5)
C(7a)-F(8)	1.345(5)	C(7a)-F(9)	1.304(4)
C(7b)-F(10)	1.325(4)	C(7b)-F(11)	1.317(4)
C(7b)-F(12)	1.333(4)		

TABLE 3b Bond angles (deg.)

N(2x)-C(1x)-C(1xa)	106.0(3)	N(2x)-C(1x)-N(4xa)	111.3(2)
C(1xa)-C(1x)-N(4xa)	104.3(3)	C(1x)-N(2x)-C(3x)	110.7(2)
N(2x)-C(3x)-N(4x)	104.9(2)	N(2x)-C(3x)-C(3ax)	108.4(2)
N(4x)-C(3x)-C(3ax)	108.2(2)	N(2x)-C(3x)-C(3bx)	112.1(3)
N(4x)-C(3x)-C(3bx)	111.8(2)	C(3ax)-C(3x)-C(3bx)	111.1(3)
C(3x)-N(4x)-C(1xa)	110.3(2)	C(3x)-C(3ax)-F(1x)	112.5(3)
C(3x)-C(3ax)-F(2x)	112.5(3)	F(1x)-C(3ax)-F(2x)	107.3(3)
C(3x)-C(3ax)-F(3x)	110.8(3)	F(1x)-C(3ax)-F(3x)	105.8(3)
F(2x)-C(3ax)-F(3x)	107.6(3)	C(3x)-C(3bx)-F(4x)	113.0(3)
C(3x)-C(3bx)-F(5x)	112.7(3)	F(4x)-C(3bx)-F(5x)	107.1(3)
C(3x)-C(3bx)-F(6x)	110.0(3)	F(4x)-C(3bx)-F(6x)	106.9(3)
F(5x)-C(3bx)-F(6x)	106.7(3)	N(2)-C(1)-C(5)	107.3(2)
N(2)-C(1)-N(8)	111.1(2)	C(5)-C(1)-N(8)	104.8(2)
C(1)-N(2)-C(3)	109.8(2)	N(2)-C(3)-N(4)	105.5(2)
N(2)-C(3)-C(3a)	108.0(2)	N(4)-C(3)-C(3a)	109.5(2)
N(2)-C(3)-C(3b)	110.4(2)	N(4)-C(3)-C(3b)	111.9(2)
C(3a)-C(3)-C(3b)	111.3(3)	C(3)-N(4)-C(5)	109.4(2)
C(1)-C(5)-N(4)	102.6(2)	C(1)-C(5)-N(6)	105.7(2)
N(4)-C(5)-N(6)	114.1(2)	C(5)-N(6)-C(7)	110.1(2)
N(6)-C(7)-N(8)	107.4(2)	N(6)-C(7)-C(7a)	110.1(3)
N(8)-C(7)-C(7a)	110.9(3)	N(6)-C(7)-C(7b)	110.9(3)
N(8)-C(7)-C(7b)	107.2(2)	C(7a)-C(7)-C(7b)	110.2(3)
C(1)-N(8)-C(7)	107.0(2)	C(3)-C(3a)-F(1)	113.4(3)
C(3)-C(3a)-F(2)	112.0(3)	F(1)-C(3a)-F(2)	107.0(3)
C(3)-C(3a)-F(3)	110.8(3)	F(1)-C(3a)-F(3)	106.7(3)
F(2)-C(3a)-F(3)	106.5(3)	C(3)-C(3b)-F(4)	113.6(3)
C(3)-C(3b)-F(5)	112.6(3)	F(4)-C(3b)-F(5)	107.4(3)
C(3)-C(3b)-F(6)	109.2(3)	F(4)-C(3b)-F(6)	107.1(3)
F(5)-C(3b)-F(6)	106.5(3)	C(7)-C(7a)-F(7)	113.7(3)
C(7)-C(7a)-F(8)	108.9(3)	F(7)-C(7a)-F(8)	105.8(3)
C(7)-C(7a)-F(9)	113.1(3)	F(7)-C(7a)-F(9)	108.9(3)
F(8)-C(7a)-F(9)	105.8(3)	C(7)-C(7b)-F(10)	112.4(3)
C(7)-C(7b)-F(11)	109.9(3)	F(10)-C(7b)-F(11)	107.4(3)
C(7)-C(7b)-F(12)	113.1(3)	F(10)-C(7b)-F(12)	106.4(3)
F(11)-C(7b)-F(12)	107.4(3)		

TABLE 4b Anisotropic temperature factors ($A^2 x 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1x)	46(2)	33(2)	52(2)	-8(1)	1(1)	-2(2)
N(2x)	41(1)	57(2)	46(1)	-7(1)	-2(1)	5(1)
C(3x)	37(2)	44(2)	44(2)	1(1)	0(1)	-5(1)
N(4x)	48(2)	65(2)	44(1)	8(1)	1(1)	-0(1)
C(3ax)	50(2)	50(2)	54(2)	-7(2)	4(2)	-4(2)
C(3bx)	50(2)	59(2)	64(2)	2(2)	3(2)	-8(2)
F(1x)	72(1)	62(1)	82(1)	-13(1)	-15(1)	21(1)
F(2x)	71(1)	87(1)	61(1)	-29(1)	-0(1)	-1(1)
F(3x)	90(2)	47(1)	114(2)	-4(1)	29(1)	-17(1)
F(4x)	45(1)	90(2)	104(2)	9(1)	-5(1)	-17(1)
F(5x)	89(2)	117(2)	70(1)	16(1)	18(1)	-33(1)
F(6x)	87(2)	43(1)	161(2)	8(1)	22(2)	-11(1)
C(1)	43(2)	34(2)	43(2)	3(1)	-0(1)	-6(1)
N(2)	40(1)	41(1)	38(1)	-3(1)	1(1)	-9(1)
C(3)	46(2)	40(2)	40(2)	-1(1)	3(1)	-5(2)
N(4)	46(1)	60(2)	40(1)	-11(1)	-2(1)	-0(1)
C(5)	40(2)	38(2)	47(2)	-5(1)	-5(1)	-8(1)
N(6)	45(1)	48(2)	40(1)	-0(1)	-3(1)	3(1)
C(7)	42(2)	41(2)	41(2)	1(1)	-1(1)	-2(1)
N(8)	45(1)	51(2)	37(1)	1(1)	1(1)	0(1)
C(3a)	89(3)	56(2)	57(2)	5(2)	9(2)	-22(2)
C(3b)	54(2)	63(2)	51(2)	-7(2)	4(2)	4(2)
C(7a)	73(2)	46(2)	58(2)	-3(2)	5(2)	-1(2)
C(7b)	50(2)	61(2)	61(2)	4(2)	2(2)	1(2)
F(1)	113(2)	159(3)	105(2)	29(2)	-13(2)	-97(2)
F(2)	142(2)	95(2)	56(1)	10(1)	32(1)	-34(2)
F(3)	174(3)	48(1)	106(2)	18(1)	28(2)	-13(2)
F(4)	62(1)	140(2)	87(1)	-31(1)	-20(1)	34(1)
F(5)	68(1)	82(1)	57(1)	-22(1)	1(1)	14(1)
F(6)	143(2)	43(1)	102(2)	7(1)	46(2)	16(1)
F(7)	126(2)	67(1)	64(1)	-26(1)	-0(1)	-5(1)
F(8)	100(2)	58(1)	123(2)	-14(1)	32(1)	-35(1)
F(9)	125(2)	49(1)	92(2)	-2(1)	-11(1)	26(1)
F(10)	70(1)	118(2)	60(1)	-3(1)	21(1)	-10(1)
F(11)	75(1)	71(1)	110(2)	-3(1)	27(1)	-30(1)
F(12)	46(1)	139(2)	103(2)	30(2)	-2(1)	8(1)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2 (h^2 a^{*2} U_{11} + k^2 b^{*2} U_{22} + \ldots + 2hka^* b^* U_{12})$$

TABLE 5b Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x 10^3$)

atom	x/a	y/b	z/c	U_{eq}
H(1x)	6850(22)	-735(19)	7134(9)	36(7)
H(2x)	5308(29)	1202(45)	7254(11)	83(10)
H(4x)	7042(27)	783(41)	8389(10)	65(9)
H(1)	9247(22)	4213(17)	9569(8)	37(7)
H(2)	10568(27)	7460(41)	9393(10)	70(9)
H(4)	9216(27)	4758(41)	8293(10)	71(9)
H(5)	7889(19)	4498(23)	8862(9)	35(7)
H(6)	6919(24)	7275(37)	8853(9)	52(8)
H(8)	8435(25)	6175(39)	10078(9)	57(8)

TABLE 1C Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
C(1)	9012(3)	3079(8)	2432(6)	50(2)*
N(2)	8296(2)	3212(5)	2433(5)	49(2)*
C(3)	7956(3)	1515(7)	2074(6)	47(2)*
N(4)	8483(3)	303(7)	2293(7)	66(2)*
C(5)	9103(3)	1040(8)	2570(6)	51(2)*
N(6)	9304(3)	941(7)	3909(6)	52(2)*
C(7)	9565(3)	2632(8)	4379(7)	49(2)*
N(8)	9336(4)	3865(7)	3462(8)	82(3)*
N(2')	8025(3)	4888(6)	2107(7)	60(2)*
O(2a)	8426(3)	6151(6)	2076(7)	82(2)*
O(2b)	7433(3)	5022(7)	1931(8)	98(3)*
N(6')	9538(3)	-731(8)	4286(7)	70(2)*
O(6a)	9424(3)	-1990(5)	3589(6)	77(2)*
O(6b)	9811(4)	-816(8)	5286(7)	107(3)*
C(3a)	7354(4)	1042(11)	2881(9)	80(3)*
C(3b)	7766(4)	1384(11)	685(8)	70(3)*
C(7a)	9254(5)	3136(15)	5640(10)	100(4)*
C(7b)	10332(4)	2760(11)	4425(10)	78(3)*
F(1)	6830(4)	1977(8)	2696(13)	218(7)*
F(2)	7186(2)	-668(6)	2737(5)	88(2)*
F(3)	7545(5)	1186(11)	4068(7)	171(4)*
F(4)	7228(4)	2193(10)	379(8)	153(3)*
F(5)	7727(3)	-281(7)	336(5)	101(2)*
F(6)	8256(5)	2161(12)	49(6)	164(4)*
F(7)	9392(4)	4815(8)	5931(7)	136(3)*
F(8)	8596(4)	3019(12)	5546(10)	186(5)*
F(9)	9437(7)	2121(11)	6538(8)	202(6)*
F(10)	10529(3)	4399(7)	4413(8)	118(3)*
F(11)	10560(3)	1954(10)	3459(11)	173(4)*
F(12)	10601(3)	2047(8)	5397(9)	151(4)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2C Bond lengths (A)

C(1)-N(2)	1.440(8)	C(1)-C(5)	1.517(9)
C(1)-N(8)	1.414(10)	N(2)-C(3)	1.474(7)
N(2)-N(2')	1.393(6)	C(3)-N(4)	1.403(8)
C(3)-C(3a)	1.532(11)	C(3)-C(3b)	1.535(11)
N(4)-C(5)	1.390(8)	C(5)-N(6)	1.307(9)
N(6)-C(7)	1.443(8)	N(6)-N(6')	1.378(8)
C(7)-N(8)	1.421(10)	C(7)-C(7a)	1.547(13)
C(7)-C(7b)	1.542(9)	N(2')-O(2a)	1.230(7)
N(2')-O(2b)	1.206(9)	N(6')-O(6a)	1.216(8)
N(6')-O(6b)	1.216(11)	C(3a)-F(1)	1.272(11)
C(3a)-F(2)	1.311(9)	C(3a)-F(3)	1.347(13)
C(3b)-F(4)	1.277(11)	C(3b)-F(5)	1.283(9)
C(3b)-F(6)	1.331(12)	C(7a)-F(7)	1.304(12)
C(7a)-F(8)	1.327(12)	C(7a)-F(9)	1.279(14)
C(7b)-F(10)	1.269(9)	C(7b)-F(11)	1.287(14)
C(7b)-F(12)	1.294(13)		

TABLE 3C Bond angles (deg.)

N(2)-C(1)-C(5)	100.8(4)	N(2)-C(1)-N(8)	115.4(6)
C(5)-C(1)-N(8)	105.7(5)	C(1)-N(2)-C(3)	113.8(4)
C(1)-N(2)-N(2')	116.8(4)	C(3)-N(2)-N(2')	120.0(5)
N(2)-C(3)-N(4)	98.3(5)	N(2)-C(3)-C(3a)	114.0(6)
N(4)-C(3)-C(3a)	110.7(6)	N(2)-C(3)-C(3b)	115.0(5)
N(4)-C(3)-C(3b)	108.0(6)	C(3a)-C(3)-C(3b)	110.1(6)
C(3)-N(4)-C(5)	117.6(5)	C(1)-C(5)-N(4)	104.9(5)
C(1)-C(5)-N(6)	100.1(5)	N(4)-C(5)-N(6)	115.4(6)
C(5)-N(6)-C(7)	113.3(5)	C(5)-N(6)-N(6')	114.8(5)
C(7)-N(6)-N(6')	122.7(6)	N(6)-C(7)-N(8)	100.7(6)
N(6)-C(7)-C(7a)	111.8(6)	N(8)-C(7)-C(7a)	109.5(6)
N(6)-C(7)-C(7b)	115.3(5)	N(8)-C(7)-C(7b)	107.9(6)
C(7a)-C(7)-C(7b)	111.0(7)	C(1)-N(8)-C(7)	116.1(5)
N(2)-N(2')-O(2a)	114.8(6)	N(2)-N(2')-O(2b)	119.9(5)
O(2a)-N(2')-O(2b)	125.3(5)	N(6)-N(6')-O(6a)	115.5(7)
N(6)-N(6')-O(6b)	117.6(6)	O(6a)-N(6')-O(6b)	126.8(6)
C(3)-C(3a)-F(1)	116.1(8)	C(3)-C(3a)-F(2)	110.7(7)
F(1)-C(3a)-F(2)	106.6(7)	C(3)-C(3a)-F(3)	107.6(7)
F(1)-C(3a)-F(3)	110.1(10)	F(2)-C(3a)-F(3)	105.2(8)
C(3)-C(3b)-F(4)	115.5(7)	C(3)-C(3b)-F(5)	111.1(6)
F(4)-C(3b)-F(5)	108.4(7)	C(3)-C(3b)-F(6)	107.0(6)
F(4)-C(3b)-F(6)	106.9(8)	F(5)-C(3b)-F(6)	107.5(7)
C(7)-C(7a)-F(7)	110.7(8)	C(7)-C(7a)-F(8)	108.5(9)
F(7)-C(7a)-F(8)	106.9(9)	C(7)-C(7a)-F(9)	114.6(9)
F(7)-C(7a)-F(9)	107.9(9)	F(8)-C(7a)-F(9)	107.9(11)
C(7)-C(7b)-F(10)	111.7(6)	C(7)-C(7b)-F(11)	107.5(7)
F(10)-C(7b)-F(11)	108.6(8)	C(7)-C(7b)-F(12)	114.7(8)
F(10)-C(7b)-F(12)	105.2(8)	F(11)-C(7b)-F(12)	109.0(8)

TABLE 4C Anisotropic temperature factors ($A^2 x 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	56(3)	38(3)	56(4)	11(3)	-7(3)	-4(3)
N(2)	54(3)	19(2)	74(3)	-1(2)	-10(3)	-2(2)
C(3)	58(4)	26(2)	57(4)	2(3)	-7(3)	-7(2)
N(4)	64(3)	39(3)	96(5)	-10(3)	-29(4)	3(2)
C(5)	55(4)	40(3)	59(4)	-5(3)	-10(3)	11(3)
N(6)	64(3)	42(3)	51(3)	6(2)	-17(3)	2(2)
C(7)	48(3)	37(3)	61(4)	-4(3)	-12(3)	3(3)
N(8)	116(6)	28(3)	104(5)	1(3)	-58(5)	-12(3)
N(2')	65(3)	29(2)	84(4)	3(3)	-22(3)	9(2)
O(2a)	91(4)	35(2)	121(5)	12(3)	-20(4)	-3(2)
O(2b)	96(4)	45(3)	153(6)	3(4)	-33(5)	22(3)
N(6')	82(4)	42(3)	85(5)	15(3)	-24(4)	-10(3)
O(6a)	104(4)	24(2)	103(4)	-1(3)	-35(3)	1(2)
O(6b)	151(6)	60(3)	112(5)	15(3)	-74(5)	-0(3)
C(3a)	74(5)	52(4)	112(7)	-4(4)	30(5)	-10(4)
C(3b)	74(5)	64(4)	72(5)	3(4)	-15(4)	-11(4)
C(7a)	92(6)	107(8)	102(7)	-48(6)	-8(6)	9(5)
C(7b)	64(4)	63(4)	108(7)	-1(5)	-9(5)	-11(4)
F(1)	116(5)	100(4)	438(19)	62(8)	146(9)	23(4)
F(2)	95(3)	69(3)	100(3)	4(3)	-3(3)	-36(2)
F(3)	265(10)	156(6)	93(4)	-43(5)	80(6)	-119(7)
F(4)	187(7)	127(5)	145(6)	-28(5)	-108(6)	42(5)
F(5)	157(5)	72(3)	75(3)	-24(3)	-33(3)	-12(3)
F(6)	240(9)	191(7)	60(3)	7(4)	-10(4)	-108(7)
F(7)	214(7)	82(4)	113(5)	-55(4)	-25(5)	13(4)
F(8)	110(5)	188(7)	260(11)	-133(8)	75(6)	-26(5)
F(9)	383(16)	126(6)	97(5)	20(5)	78(7)	90(8)
F(10)	98(3)	74(3)	181(6)	29(4)	-40(4)	-46(3)
F(11)	88(4)	179(7)	253(10)	-107(7)	66(5)	-57(4)
F(12)	97(4)	116(5)	239(10)	72(6)	-85(5)	-19(3)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$$

TABLE 5C Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x 10^3$)

atom	x/a	y/b	z/c	U
H(1)	9174(34)	3699(85)	1712(39)	59
H(4)	8390(37)	-754(31)	2230(87)	77
H(5)	9473(22)	579(67)	2102(57)	60
H(8)	9144(44)	4668(96)	3789(89)	98

TABLE 1d Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
C(1)	-1858(4)	7515(2)	1347(3)	42(1)*
N(2)	-1761(4)	6931(1)	2120(2)	58(1)*
C(3)	-455(5)	6360(2)	1941(3)	50(1)*
N(4)	616(4)	6702(1)	1122(2)	48(1)*
C(5)	-22(4)	7403(2)	807(2)	40(1)*
N(6)	1210(3)	7943(1)	1353(2)	42(1)*
C(7)	116(4)	8509(2)	1793(3)	44(1)*
N(8)	-1695(4)	8175(1)	1867(3)	61(1)*
N(2')	-3083(5)	6918(2)	2898(3)	63(1)*
O(2a)	-4382(4)	7331(2)	2769(2)	74(1)*
O(2b)	-2800(5)	6509(2)	3636(3)	93(1)*
N(4')	1359(5)	6286(2)	280(3)	67(1)*
O(4a)	1985(5)	5722(2)	574(3)	98(1)*
O(4b)	1259(4)	6531(2)	-621(2)	75(1)*
N(6')	3004(4)	8035(2)	995(3)	56(1)*
O(6a)	3570(4)	7564(2)	452(2)	67(1)*
O(6b)	3897(4)	8549(2)	1328(3)	87(1)*
C(3a)	941(6)	6194(2)	2976(3)	65(1)*
C(3b)	-1644(6)	5698(2)	1487(3)	65(1)*
C(7a)	957(6)	8732(2)	2970(3)	62(1)*
C(7b)	-169(5)	9159(2)	1036(3)	55(1)*
F(1)	280(4)	5697(2)	3580(2)	94(1)*
F(2)	2613(4)	5968(2)	2703(2)	91(1)*
F(3)	1265(4)	6757(1)	3566(2)	92(1)*
F(4)	-3214(4)	5635(1)	1989(2)	92(1)*
F(5)	-684(4)	5120(1)	1629(2)	98(1)*
F(6)	-2166(4)	5798(1)	451(2)	85(1)*
F(7)	-336(6)	9140(2)	3372(3)	86(1)*
F(8)	1001(11)	8159(2)	3604(3)	127(3)*
F(9)	2595(6)	9029(4)	3044(3)	125(2)*
F(10)	-1690(12)	9520(5)	1251(10)	78(3)*
F(11)	-476(11)	8980(2)	35(3)	100(2)*
F(12)	1254(9)	9609(3)	1140(9)	111(3)*
F(7')	-277(12)	8956(6)	3556(7)	81(3)*
F(8')	2012(12)	8240(4)	3407(5)	71(3)*
F(9')	2110(11)	9298(4)	2908(6)	66(3)*
F(10')	-1182(34)	9633(13)	1482(25)	132(9)*
F(11')	-1175(14)	8898(4)	166(6)	89(3)*
F(12')	1421(22)	9387(7)	688(8)	77(4)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2d Bond lengths (A)

C(1)-N(2)	1.464(4)	C(1)-C(5)	1.517(4)
C(1)-N(8)	1.414(4)	N(2)-C(3)	1.453(4)
N(2)-N(2')	1.395(3)	C(3)-N(4)	1.467(4)
C(3)-C(3a)	1.565(5)	C(3)-C(3b)	1.566(5)
N(4)-C(5)	1.451(4)	N(4)-N(4')	1.444(4)
C(5)-N(6)	1.467(4)	N(6)-C(7)	1.459(4)
N(6)-N(6')	1.381(4)	C(7)-N(8)	1.429(4)
C(7)-C(7a)	1.573(4)	C(7)-C(7b)	1.555(4)
N(2')-O(2a)	1.203(4)	N(2')-O(2b)	1.202(5)
N(4')-O(4a)	1.206(4)	N(4')-O(4b)	1.202(4)
N(6')-O(6a)	1.210(4)	N(6')-O(6b)	1.214(4)
C(3a)-F(1)	1.317(5)	C(3a)-F(2)	1.320(5)
C(3a)-F(3)	1.306(5)	C(3b)-F(4)	1.316(5)
C(3b)-F(5)	1.296(4)	C(3b)-F(6)	1.310(4)
C(7a)-F(7)	1.325(5)	C(7a)-F(8)	1.344(5)
C(7a)-F(9)	1.274(5)	C(7a)-F(7')	1.253(10)
C(7a)-F(8')	1.281(9)	C(7a)-F(9')	1.355(8)
C(7b)-F(10)	1.316(10)	C(7b)-F(11)	1.281(5)
C(7b)-F(12)	1.312(7)	C(7b)-F(10')	1.303(26)
C(7b)-F(11')	1.325(8)	C(7b)-F(12')	1.304(15)

Table 3d Bond angles (deg.)

N(2)-C(1)-C(5)	100.9(2)	N(2)-C(1)-N(8)	112.7(3)
C(5)-C(1)-N(8)	106.8(2)	C(1)-N(2)-C(3)	117.6(3)
C(1)-N(2)-N(2')	118.0(3)	C(3)-N(2)-N(2')	123.9(3)
N(2)-C(3)-N(4)	98.0(2)	N(2)-C(3)-C(3a)	112.4(3)
N(4)-C(3)-C(3a)	109.4(3)	N(2)-C(3)-C(3b)	109.6(3)
N(4)-C(3)-C(3b)	113.3(3)	C(3a)-C(3)-C(3b)	113.1(3)
C(3)-N(4)-C(5)	115.5(2)	C(3)-N(4)-N(4')	105.3(2)
C(1)-C(5)-N(6)	100.6(2)	N(4)-C(5)-N(6)	111.9(2)
C(5)-N(6)-C(7)	112.7(2)	C(5)-N(6)-N(6')	117.0(2)
C(7)-N(6)-N(6')	123.4(2)	N(6)-C(7)-N(8)	101.1(2)
N(6)-C(7)-C(7a)	112.3(2)	N(8)-C(7)-C(7a)	108.6(3)
N(6)-C(7)-C(7b)	114.2(3)	N(8)-C(8)-C(7b)	109.3(3)
C(7a)-C(7)-C(7b)	110.8(3)	C(1)-N(8)-C(7)	113.8(3)
N(2)-N(2')-O(2a)	116.0(3)	N(2)-N(2')-O(2b)	117.4(3)
O(2a)-N(2')-O(2b)	126.5(4)	N(4)-N(4')-O(4a)	114.8(3)
N(4)-N(4')-O(4b)	117.3(3)	O(4a)-N(4')-O(4b)	127.9(4)
N(6)-N(6')-O(6a)	115.9(3)	N(6)-N(6')-O(6b)	116.6(3)
O(6a)-N(6')-O(6b)	127.1(3)	C(3)-C(3a)-F(1)	112.5(3)
C(3)-C(3a)-F(2)	111.0(3)	F(1)-C(3a)-F(2)	106.0(3)
C(3)-C(3a)-F(3)	110.5(3)	F(1)-C(3a)-F(3)	109.3(3)
F(2)-C(3a)-F(3)	107.4(3)	C(3)-C(3b)-F(4)	109.8(3)
C(3)-C(3b)-F(5)	112.5(3)	F(4)-C(3b)-F(5)	107.7(3)
C(3)-C(3b)-F(6)	108.5(3)	F(4)-C(3b)-F(6)	107.7(3)
F(5)-C(3b)-F(6)	110.5(3)	C(7)-C(7a)-F(7)	107.2(3)
C(7)-C(7a)-F(8)	107.7(3)	F(7)-C(7a)-F(8)	103.9(4)
C(7)-C(7a)-F(9)	116.3(3)	F(7)-C(7a)-F(9)	110.6(4)
F(8)-C(7a)-F(9)	110.4(5)	C(7)-C(7a)-C(7')	114.0(5)
C(7)-C(7a)-F(9')	109.7(4)	F(8')-C(7a)-F(9')	106.6(6)
F(7')-C(7a)-F(9')	101.8(7)	C(7)-C(7b)-F(11)	111.5(3)
C(7)-C(7b)-F(10)	110.9(6)	C(7)-C(7b)-F(12)	114.3(5)
F(10)-C(7b)-F(11)	105.6(7)	F(11)-C(7b)-F(12)	109.0(5)
F(10)-C(7b)-F(12)	105.0(6)	C(7)-C(7b)-F(12')	113.6(5)
C(7)-C(7b)-F(10')	110.2(13)	F(10')-C(7b)-F(12')	114.9(13)
C(7)-C(7b)-C(11')	102.4(4)		
F(10')-C(7b)-F(11')	109.6(12)		
F(11')-C(7b)-F(12')	105.3(7)		

TABLE 4d Anisotropic temperature factors ($A^2 \times 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	40(2)	40(2)	46(2)	0(1)	3(1)	-4(1)
N(2)	69(2)	47(1)	61(2)	11(1)	30(1)	10(1)
C(3)	61(2)	36(2)	52(2)	3(1)	1(2)	1(1)
N(4)	58(2)	39(1)	49(1)	-2(1)	16(1)	2(1)
C(5)	39(1)	39(1)	41(2)	2(1)	2(1)	4(1)
N(6)	31(1)	40(1)	56(1)	0(1)	8(1)	1(1)
C(7)	45(2)	33(1)	53(2)	-4(1)	5(1)	2(1)
N(8)	47(2)	42(1)	99(2)	-14(2)	27(2)	-6(1)
N(2')	68(2)	68(2)	56(2)	3(2)	21(2)	-15(2)
O(2a)	66(2)	78(2)	81(2)	-4(2)	30(1)	-4(2)
O(2b)	104(2)	101(2)	77(2)	24(2)	34(2)	-13(2)
N(4')	72(2)	55(2)	74(2)	-18(2)	20(2)	7(2)
O(4a)	136(3)	61(2)	97(2)	-1(2)	23(2)	50(2)
O(4b)	98(2)	79(2)	52(1)	-8(1)	23(1)	11(2)
N(6')	41(1)	61(2)	68(2)	6(2)	8(1)	-4(1)
O(6a)	51(1)	81(2)	72(2)	-11(1)	21(1)	7(1)
O(6b)	58(2)	69(2)	136(3)	-19(2)	25(2)	-27(1)
C(3a)	81(3)	57(2)	54(2)	5(2)	-6(2)	-1(2)
C(3b)	83(3)	46(2)	64(2)	4(2)	-4(2)	-10(2)
C(7a)	80(3)	60(2)	45(2)	-4(2)	6(2)	3(2)
C(7b)	63(2)	43(2)	60(2)	7(2)	5(2)	4(2)
F(1)	112(2)	91(2)	75(1)	35(2)	-7(1)	-7(2)
F(2)	78(2)	95(2)	95(2)	9(2)	-16(1)	17(1)
F(3)	119(2)	82(2)	70(1)	-12(1)	-21(1)	-9(1)
F(4)	97(2)	81(2)	98(2)	11(2)	11(1)	-38(1)
F(5)	135(2)	42(1)	111(2)	-2(1)	-12(2)	-2(1)
F(6)	111(2)	78(2)	52(1)	-3(1)	-20(1)	-25(1)
F(7)	133(3)	66(2)	63(2)	-22(2)	30(2)	5(2)
F(8)	255(7)	72(2)	53(2)	9(2)	8(3)	38(3)
F(9)	67(2)	222(6)	82(2)	-59(3)	-11(2)	-22(3)
F(10)	84(3)	60(3)	93(6)	8(3)	22(3)	32(2)
F(11)	165(5)	80(2)	55(2)	12(2)	19(3)	54(3)
F(12)	85(3)	52(3)	191(8)	43(4)	-19(4)	-22(2)
F(7')	74(5)	109(7)	62(5)	-12(5)	11(4)	-27(5)
F(8')	85(6)	87(6)	38(4)	3(4)	-14(3)	-2(5)
F(9')	64(4)	52(4)	78(5)	-17(4)	-12(4)	-29(4)
F(10')	231(24)	77(11)	80(7)	-22(8)	-25(14)	106(14)
F(11')	115(7)	80(5)	60(4)	-4(4)	-51(4)	6(5)
F(12')	122(8)	57(7)	53(5)	-7(4)	15(5)	-31(6)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+ \ldots +2hka^*b^*U_{12})$$

TABLE 5d Hydrogen coordinates ($\times 10^4$) and temperature factors ($A^2 \times 10^3$)

atom	x/a	y/b	z/c	U
H(1)	-3114(55)	7508(19)	960(29)	65(11)
H(5)	-177(38)	7451(13)	-32(21)	29(7)
H(8)	-3041(40)	8500(15)	1854(22)	47(8)

TABLE 18 Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
C(1)	3569(1)	6743(3)	3424(1)	34(1)*
N(2)	3744(1)	8556(2)	3387(1)	33(1)*
C(3)	4187(1)	8534(3)	3647(1)	33(1)*
N(4)	4248(1)	6627(2)	3549(1)	36(1)*
C(5)	3894(1)	5510(3)	3156(1)	34(1)*
N(6)	3756(1)	5666(3)	2060(1)	42(1)*
C(7)	3322(1)	5868(3)	1711(2)	40(1)*
N(8)	3225(1)	6511(3)	2632(1)	42(1)*
N(2')	3536(1)	9965(2)	3745(1)	44(1)*
O(2a)	3199(1)	9611(3)	3828(2)	55(1)*
O(2b)	3706(1)	11406(2)	3890(1)	57(1)*
N(4')	4634(1)	5804(3)	3709(2)	48(1)*
O(4a)	4911(1)	6794(3)	3656(2)	63(1)*
O(4b)	4641(1)	4236(2)	3896(2)	68(1)*
N(6')	4010(1)	5167(2)	1445(1)	46(1)*
O(6a)	4334(1)	4604(2)	1853(1)	59(1)*
O(6b)	3884(1)	5390(3)	560(1)	64(1)*
N(8')	2844(1)	6175(3)	2864(2)	53(1)*
O(8a)	2571(1)	5933(4)	2164(2)	80(1)*
O(8b)	2832(1)	6221(3)	3720(1)	64(1)*
C(3a)	4360(1)	9681(3)	2878(2)	43(1)*
C(3b)	4355(1)	9117(3)	4762(2)	40(1)*
C(7a)	3201(1)	7323(3)	891(2)	51(1)*
C(7b)	3131(1)	3946(3)	1385(2)	50(1)*
F(1)	4121(1)	11061(2)	2544(1)	62(1)*
F(2)	4720(1)	10299(2)	3243(1)	62(1)*
F(3)	4373(1)	8670(2)	2093(1)	57(1)*
F(4)	4447(1)	10828(2)	4861(1)	59(1)*
F(5)	4680(1)	8190(2)	5160(1)	61(1)*
F(6)	4079(1)	8745(2)	5277(1)	49(1)*
F(7)	2864(1)	8142(3)	980(1)	83(1)*
F(8)	3488(1)	8562(2)	1001(1)	68(1)*
F(9)	3141(1)	6670(2)	-6(1)	70(1)*
F(10)	2774(1)	4051(2)	786(1)	67(1)*
F(11)	3101(1)	3041(2)	2186(1)	71(1)*
F(12)	3367(1)	3027(2)	929(1)	69(1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2e Bond lengths (A)

C(1)-N(2)	1.482(3)	C(1)-C(5)	1.501(3)
C(1)-N(8)	1.435(2)	N(2)-C(3)	1.475(2)
N(2)-N(2')	1.417(2)	C(3)-N(4)	1.448(2)
C(3)-C(3a)	1.577(3)	C(3)-C(3b)	1.591(3)
N(4)-C(5)	1.431(2)	N(4)-N(4')	1.427(2)
C(5)-N(6)	1.493(2)	N(6)-C(7)	1.462(2)
N(6)-N(6')	1.393(3)	C(7)-N(8)	1.467(3)
C(7)-C(7a)	1.562(3)	C(7)-C(7b)	1.598(3)
N(8)-N(8')	1.425(3)	N(2')-O(2a)	1.207(3)
N(2')-O(2b)	1.217(2)	N(4')-O(4a)	1.211(3)
N(4')-O(4b)	1.197(3)	N(6')-O(6a)	1.203(2)
N(6')-O(6b)	1.222(2)	N(8')-O(8a)	1.205(2)
N(8')-O(8b)	1.197(3)	C(3a)-F(1)	1.331(2)
C(3a)-F(2)	1.306(2)	C(3a)-F(3)	1.333(3)
C(3b)-F(4)	1.314(2)	C(3b)-F(5)	1.322(2)
C(3b)-F(6)	1.325(3)	C(7a)-F(7)	1.328(3)
C(7a)-F(8)	1.330(3)	C(7a)-F(9)	1.312(3)
C(7b)-F(10)	1.321(3)	C(7b)-F(11)	1.322(3)
C(7b)-F(12)	1.319(3)		

TABLE 3e Bond angles (deg.)

N(2)-C(1)-C(5)	100.4(1)	N(2)-C(1)-N(8)	110.6(2)
C(5)-C(1)-N(8)	105.5(2)	C(1)-N(2)-C(3)	112.4(1)
C(1)-N(2)-N(2')	115.3(2)	C(3)-N(2)-N(2')	119.1(1)
N(2)-C(3)-N(4)	98.5(1)	N(2)-C(3)-C(3a)	109.9(1)
N(4)-C(3)-C(3a)	112.8(2)	N(2)-C(3)-C(3b)	111.7(2)
N(4)-C(3)-C(3b)	109.3(1)	C(3a)-C(3)-C(3b)	113.7(2)
C(3)-N(4)-C(5)	115.5(1)	C(3)-N(4)-N(4')	123.7(1)
C(5)-N(4)-N(4')	120.4(2)	C(1)-C(5)-N(4)	102.3(1)
C(1)-C(5)-N(6)	98.6(1)	N(4)-C(5)-N(6)	115.4(2)
C(5)-N(6)-C(7)	114.5(2)	C(5)-N(6)-N(6')	121.1(1)
C(7)-N(6)-N(6')	122.9(2)	N(6)-C(7)-N(8)	98.4(1)
N(6)-C(7)-C(7a)	113.7(2)	N(8)-C(7)-C(7a)	109.5(2)
N(6)-C(7)-C(7b)	108.8(2)	N(8)-C(7)-C(7b)	112.7(2)
C(7a)-C(7)-C(7b)	112.9(2)	C(1)-N(8)-C(7)	114.0(2)
C(1)-N(8)-N(8')	118.8(2)	C(7)-N(8)-N(8')	121.7(2)
N(2)-N(2')-O(2a)	115.5(2)	N(2)-N(2')-O(2b)	117.2(2)
O(2a)-N(2')-O(2b)	127.3(2)	N(4)-N(4')-O(4a)	115.6(2)
N(4)-N(4')-O(4b)	115.2(2)	O(4a)-N(4')-O(4b)	129.1(2)
N(6)-N(6')-O(6a)	115.7(2)	N(6)-N(6')-O(6b)	116.7(2)
O(6a)-N(6')-O(6b)	127.6(2)	N(8)-N(8')-O(8a)	115.2(2)
N(8)-N(8')-O(8b)	116.4(2)	O(8a)-N(8')-O(8b)	128.3(2)
C(3)-C(3a)-F(1)	111.4(2)	C(3)-C(3a)-F(2)	112.6(2)
F(1)-C(3a)-F(2)	108.3(2)	C(3)-C(3a)-F(3)	109.3(2)
F(1)-C(3a)-F(3)	106.1(2)	F(2)-C(3a)-F(3)	108.9(2)
C(3)-C(3b)-F(4)	113.2(2)	C(3)-C(3b)-F(5)	110.9(2)
F(4)-C(3b)-F(5)	107.6(2)	C(3)-C(3b)-F(6)	108.6(1)
F(4)-C(3b)-F(6)	109.2(2)	F(5)-C(3b)-F(6)	107.0(2)
C(7)-C(7a)-F(7)	110.4(2)	C(7)-C(7a)-F(8)	109.1(2)
F(7)-C(7a)-F(8)	107.4(2)	C(7)-C(7a)-F(9)	113.4(2)
F(7)-C(7a)-F(9)	107.3(2)	F(8)-C(7a)-F(9)	109.1(2)
C(7)-C(7b)-F(10)	112.9(2)	C(7)-C(7b)-F(11)	108.6(2)
F(10)-C(7b)-F(11)	109.5(2)	C(7)-C(7b)-F(12)	110.4(2)
F(10)-C(7b)-F(12)	107.7(2)	F(11)-C(7b)-F(12)	107.6(2)

TABLE 4e Anisotropic temperature factors ($A^2 x 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	31(1)	42(1)	28(1)	2(1)	4(1)	-3(1)
N(2)	29(1)	34(1)	34(1)	-2(1)	3(1)	3(1)
C(3)	30(1)	31(1)	36(1)	-1(1)	5(1)	0(1)
N(4)	27(1)	30(1)	47(1)	-1(1)	2(1)	-0(1)
C(5)	33(1)	31(1)	36(1)	2(1)	4(1)	-3(1)
N(6)	35(1)	56(1)	36(1)	-11(1)	9(1)	-6(1)
C(7)	37(1)	49(1)	33(1)	-4(1)	5(1)	-9(1)
N(8)	30(1)	63(1)	32(1)	-6(1)	5(1)	-8(1)
N(2')	42(1)	44(1)	42(1)	-3(1)	1(1)	13(1)
O(2a)	39(1)	73(1)	83(1)	-16(1)	12(1)	12(1)
O(2b)	71(1)	38(1)	63(1)	-10(1)	14(1)	4(1)
N(4')	34(1)	46(1)	59(1)	-4(1)	1(1)	8(1)
O(4a)	32(1)	68(1)	90(1)	-12(1)	16(1)	-4(1)
O(4b)	59(1)	42(1)	96(1)	5(1)	-0(1)	17(1)
N(6')	52(1)	42(1)	49(1)	-13(1)	21(1)	-11(1)
O(6a)	52(1)	56(1)	72(1)	-11(1)	24(1)	3(1)
O(6b)	81(1)	74(1)	44(1)	-11(1)	25(1)	-10(1)
N(8')	31(1)	78(1)	50(1)	-4(1)	8(1)	-5(1)
O(8a)	33(1)	138(2)	62(1)	-17(1)	-3(1)	-15(1)
O(8b)	43(1)	106(1)	48(1)	3(1)	17(1)	-9(1)
C(3a)	50(1)	36(1)	43(1)	-3(1)	11(1)	-8(1)
C(3b)	37(1)	41(1)	38(1)	-3(1)	3(1)	-5(1)
C(7a)	55(1)	56(1)	40(1)	1(1)	4(1)	-10(1)
C(7b)	53(1)	54(1)	40(1)	-4(1)	5(1)	-18(1)
F(1)	84(1)	43(1)	62(1)	18(1)	22(1)	5(1)
F(2)	58(1)	66(1)	67(1)	-6(1)	21(1)	-27(1)
F(3)	77(1)	55(1)	43(1)	-7(1)	26(1)	-13(1)
F(4)	74(1)	48(1)	51(1)	-15(1)	8(1)	-19(1)
F(5)	45(1)	76(1)	52(1)	-6(1)	-14(1)	9(1)
F(6)	52(1)	63(1)	33(1)	-0(1)	9(1)	-4(1)
F(7)	74(1)	97(1)	73(1)	21(1)	7(1)	30(1)
F(8)	84(1)	51(1)	61(1)	8(1)	1(1)	-21(1)
F(9)	88(1)	80(1)	35(1)	4(1)	-0(1)	-20(1)
F(10)	58(1)	89(1)	48(1)	-9(1)	-4(1)	-32(1)
F(11)	85(1)	67(1)	56(1)	12(1)	9(1)	-29(1)
F(12)	81(1)	55(1)	74(1)	-21(1)	21(1)	-19(1)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$$

TABLE 5e Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x	y	z	U
H(1)	3534(7)	6491(33)	3987(21)	53(7)
H(5)	3903(6)	4635(27)	3408(15)	29(5)

TABLE 1f Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
N(1)	1814(5)	2572(3)	2623(5)	57(3)*
C(2)	1053(6)	3192(4)	2004(6)	64(4)*
N(3)	-304(5)	3061(3)	1682(5)	53(3)*
C(4)	-810(6)	2427(4)	890(6)	57(4)*
N(5)	-122(5)	1758(3)	1373(4)	51(3)*
N(6)	211(5)	1692(3)	2630(4)	57(3)*
C(7)	1284(7)	2166(4)	3366(6)	64(4)*
N(8)	2057(5)	2074(3)	1824(5)	60(3)*
C(9)	977(6)	1605(4)	1077(6)	60(3)*
N(10)	-1057(6)	3690(4)	1228(6)	64(3)*
C(11)	-1390(6)	4140(4)	1972(6)	59(3)*
O(11)	-1047(4)	4015(3)	3042(4)	75(2)*
C(12)	-2233(7)	4760(4)	1360(7)	60(4)*
C(13)	-2311(8)	5054(4)	260(8)	75(5)*
C(14)	-3198(11)	5603(5)	-281(9)	106(6)*
C(15)	-3976(9)	5869(5)	264(9)	109(6)*
C(16)	-3884(8)	5601(4)	1341(9)	104(6)*
C(17)	-3000(7)	5060(4)	1918(8)	83(5)*
C(18)	-517(7)	1307(4)	3111(6)	58(3)*
O(18)	-281(4)	1370(3)	4196(4)	77(2)*
C(19)	-1513(6)	822(3)	2300(5)	51(3)*
C(20)	-2672(9)	811(5)	2409(6)	70(4)*
C(21)	-3622(8)	326(6)	1744(8)	92(6)*
C(22)	-3373(8)	-166(5)	1004(8)	90(5)*
C(23)	-2231(7)	-167(4)	875(6)	78(4)*
C(24)	-1309(7)	324(4)	1520(6)	65(4)*
C(25)	3171(7)	2049(4)	1645(6)	62(4)*
O(25)	3220(5)	1619(3)	872(5)	80(3)*
C(26)	4305(7)	2499(4)	2382(6)	49(3)*
C(27)	5231(7)	2526(4)	1921(7)	86(5)*
C(28)	6331(8)	2931(5)	2490(8)	99(5)*
C(29)	6544(7)	3282(4)	3546(7)	71(4)*
C(30)	5635(7)	3243(4)	4018(7)	82(4)*
C(31)	4520(7)	2860(4)	3447(6)	75(4)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 3f Bond lengths (A)

N(1)-C(2)	1.441(8)	N(1)-C(7)	1.447(11)
N(1)-N(8)	1.416(9)	C(2)-N(3)	1.457(9)
N(3)-C(4)	1.465(8)	N(3)-N(10)	1.406(9)
C(4)-N(5)	1.442(8)	N(5)-N(6)	1.411(7)
N(5)-C(9)	1.442(10)	N(6)-C(7)	1.480(8)
N(6)-C(18)	1.361(10)	N(8)-C(9)	1.483(8)
N(8)-C(25)	1.357(11)	N(10)-C(11)	1.360(11)
C(11)-O(11)	1.210(8)	C(11)-C(12)	1.481(9)
C(12)-C(13)	1.390(13)	C(12)-C(17)	1.388(13)
C(13)-C(14)	1.391(13)	C(14)-C(15)	1.365(18)
C(15)-C(16)	1.343(15)	C(16)-C(17)	1.387(11)
C(18)-O(18)	1.228(8)	C(18)-C(19)	1.472(8)
C(19)-C(20)	1.368(13)	C(19)-C(24)	1.379(11)
C(20)-C(21)	1.388(12)	C(21)-C(22)	1.358(15)
C(22)-C(23)	1.360(13)	C(23)-C(24)	1.369(9)
C(25)-O(25)	1.226(10)	C(25)-C(26)	1.499(9)
C(26)-C(27)	1.357(13)	C(26)-C(31)	1.369(10)
C(27)-C(28)	1.385(11)	C(28)-C(29)	1.352(13)
C(29)-C(30)	1.349(14)	C(30)-C(31)	1.378(10)

TABLE 3f Bond angles (deg.)

C(2)-N(1)-C(7)	114.1(6)	C(2)-N(1)-N(8)	112.5(5)
C(7)-N(1)-N(8)	108.5(5)	N(1)-C(2)-N(3)	112.2(6)
C(2)-N(3)-C(4)	114.7(6)	C(2)-N(3)-N(10)	113.4(6)
C(4)-N(3)-N(10)	110.9(5)	N(3)-C(4)-N(5)	112.5(5)
C(4)-N(5)-N(6)	112.8(5)	C(4)-N(5)-C(9)	116.8(6)
N(6)-N(5)-C(9)	109.5(4)	N(5)-N(6)-C(7)	114.6(6)
N(5)-N(6)-C(18)	122.1(5)	C(7)-N(6)-C(18)	122.7(6)
N(1)-C(7)-N(6)	111.7(6)	N(1)-N(8)-C(9)	116.6(6)
N(1)-N(8)-C(25)	123.7(5)	C(9)-N(8)-C(25)	119.4(6)
N(5)-C(9)-N(8)	109.5(6)	N(3)-N(10)-C(11)	120.8(6)
N(10)-C(11)-O(11)	121.3(6)	N(10)-C(11)-C(12)	114.8(6)
O(11)-C(11)-C(12)	123.8(7)	C(11)-C(12)-C(13)	124.0(8)
C(11)-C(12)-C(17)	117.7(7)	C(13)-C(12)-C(17)	118.3(7)
C(12)-C(13)-C(14)	119.6(10)	C(13)-C(14)-C(15)	121.0(10)
C(14)-C(15)-C(16)	119.8(8)	C(15)-C(16)-C(17)	121.0(10)
C(12)-C(17)-C(16)	120.2(9)	N(6)-C(18)-O(18)	118.1(6)
N(6)-C(18)-C(19)	117.7(6)	O(18)-C(18)-C(19)	124.1(7)
C(18)-C(19)-C(20)	118.1(7)	C(18)-C(19)-C(24)	123.8(7)
C(20)-C(19)-C(24)	117.7(6)	C(19)-C(20)-C(21)	121.4(8)
C(20)-C(21)-C(22)	119.0(9)	C(21)-C(22)-C(23)	120.9(8)
C(22)-C(23)-C(24)	119.5(8)	C(19)-C(24)-C(23)	121.5(8)
N(8)-C(25)-O(25)	116.6(6)	N(8)-C(25)-C(26)	122.1(7)
O(25)-C(25)-C(26)	121.3(7)	C(25)-C(26)-C(27)	113.8(7)
C(25)-C(26)-C(31)	128.5(8)	C(27)-C(26)-C(31)	117.7(7)
C(26)-C(27)-C(28)	120.6(8)	C(27)-C(28)-C(29)	121.5(10)
C(28)-C(29)-C(30)	117.9(7)	C(29)-C(30)-C(31)	121.3(8)
C(26)-C(31)-C(30)	120.9(8)		

TABLE 4f Anisotropic temperature factors ($A^2 \times 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	46(4)	70(5)	53(4)	-10(4)	15(3)	-1(4)
C(2)	48(5)	72(6)	67(5)	-21(4)	15(4)	-15(5)
N(3)	50(4)	57(4)	52(4)	-11(3)	18(3)	2(4)
C(4)	56(5)	70(6)	46(5)	-5(4)	20(4)	-12(5)
N(5)	51(4)	58(4)	44(3)	1(3)	17(3)	-2(3)
N(6)	54(4)	66(4)	45(4)	-3(3)	12(3)	-12(4)
C(7)	53(5)	80(6)	64(5)	-5(5)	29(5)	-2(5)
N(8)	54(4)	64(4)	58(4)	-22(4)	18(4)	-18(4)
C(9)	73(5)	60(5)	58(5)	-11(4)	37(4)	-11(5)
N(10)	60(4)	69(5)	63(5)	-7(4)	24(4)	3(4)
C(11)	43(5)	74(6)	59(5)	-16(4)	18(4)	-7(4)
O(11)	64(3)	106(4)	56(3)	-16(3)	24(3)	-10(3)
C(12)	67(6)	44(5)	70(5)	-19(4)	28(5)	-21(4)
C(13)	83(7)	54(6)	90(7)	-8(5)	35(6)	7(5)
C(14)	138(10)	59(7)	115(9)	-12(6)	41(8)	2(7)
C(15)	97(8)	42(6)	165(10)	-5(6)	21(7)	-9(6)
C(16)	92(7)	54(6)	183(10)	-17(6)	69(7)	8(6)
C(17)	90(7)	67(6)	106(7)	-31(5)	55(6)	-16(6)
C(18)	51(5)	69(6)	58(5)	10(5)	24(4)	7(5)
O(18)	87(4)	107(4)	46(3)	-0(3)	34(3)	-7(3)
C(19)	40(4)	61(5)	53(5)	16(4)	17(4)	1(4)
C(20)	54(6)	102(8)	57(6)	-9(5)	24(5)	3(6)
C(21)	49(7)	142(10)	91(8)	15(7)	30(6)	0(7)
C(22)	85(8)	78(7)	92(7)	19(6)	17(6)	-24(6)
C(23)	103(7)	48(5)	74(5)	-2(4)	25(5)	-17(5)
C(24)	73(6)	52(5)	79(6)	4(5)	39(5)	10(5)
C(25)	56(5)	71(6)	72(5)	3(5)	40(5)	-2(5)
O(25)	92(4)	79(4)	92(4)	-30(3)	61(4)	-10(3)
C(26)	52(5)	52(5)	54(5)	-3(4)	33(4)	2(4)
C(27)	90(7)	92(7)	91(7)	-21(5)	51(6)	-17(6)
C(28)	75(7)	144(9)	99(7)	-24(7)	56(6)	-10(6)
C(29)	51(6)	89(7)	72(6)	19(5)	21(5)	-5(5)
C(30)	67(6)	96(7)	79(6)	-15(5)	24(5)	-15(5)
C(31)	65(5)	87(6)	84(6)	-18(5)	42(5)	-2(5)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2 (h^2 a^{*2} U_{11} + k^2 b^{*2} U_{22} + \ldots + 2hka^*b^* U_{12})$$

TABLE 5f Hydrogen coordinates ($\times 10^4$) and temperature factors ($A^2 \times$

atom	x/a	y/b	z/c	U
H(2a)	1298	3615	2521	90
H(2b)	1209	3284	1280	80
H(4a)	-756	2525	122	80
H(4b)	-1686	2359	786	85
H(7a)	1933	1862	3913	79
H(7b)	984	2508	3812	79
H(9a)	1207	1096	1234	80
H(9b)	779	1709	239	68
H(10)	-1114(63)	3738(37)	494(34)	75
H(13)	-1757	4878	-123	88
H(14)	-3262	5798	-1048	115
H(15)	-4587	6246	-122	116
H(16)	-4439	5786	1716	119
H(17)	-2919	4893	2705	101
H(20)	-2834	1146	2957	90
H(21)	-4444	337	1804	95
H(22)	-4012	-518	567	90
H(23)	-2370	-508	335	91
H(24)	-503	322	1424	77
H(27)	5125	2262	1193	80
H(28)	6956	2963	2128	94
H(29)	7319	3550	3948	81
H(30)	5766	3488	4766	95
H(31)	3885	2845	3799	88

TABLE 1g Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
N(1)	-42(1)	2495(1)	3183(1)	38(1)*
C(2)	-565(2)	3397(1)	2924(2)	44(1)*
N(3)	270(1)	4084(1)	2971(1)	42(1)*
C(4)	962(2)	3826(1)	2311(1)	43(1)*
N(5)	1388(1)	2894(1)	2432(1)	40(1)*
C(6)	515(2)	2250(1)	2400(1)	41(1)*
N(7)	-860(1)	1835(1)	3172(1)	44(1)*
C(8)	-946(2)	1519(1)	4090(1)	39(1)*
O(9)	-387(1)	1807(1)	4942(1)	58(1)*
C(10)	-1804(2)	798(1)	4002(2)	40(1)*
C(11)	-1666(2)	200(1)	4838(2)	52(1)*
C(12)	-2436(2)	-481(2)	4779(2)	64(1)*
C(13)	-3348(2)	-564(2)	3911(2)	67(1)*
C(14)	-3486(2)	29(2)	3094(2)	64(1)*
C(15)	-2723(2)	707(1)	3132(2)	50(1)*
N(16)	891(1)	4261(1)	4027(1)	40(1)*
C(17)	1366(2)	5083(1)	4281(1)	38(1)*
O(18)	1276(1)	5701(1)	3641(1)	60(1)*
C(19)	2046(2)	5187(1)	5398(1)	39(1)*
C(20)	2912(2)	5798(2)	5632(2)	64(1)*
C(21)	3588(2)	5899(2)	6658(2)	76(1)*
C(22)	3398(2)	5405(2)	7444(2)	69(1)*
C(23)	2524(2)	4815(2)	7226(2)	62(1)*
C(24)	1848(2)	4700(1)	6212(2)	49(1)*
N(25)	2305(1)	2777(1)	3329(1)	44(1)*
C(26)	3290(2)	2576(1)	3196(1)	40(1)*
O(27)	3422(1)	2459(1)	2331(1)	60(1)*
C(28)	4229(2)	2508(1)	4184(2)	42(1)*
C(29)	5107(2)	1944(2)	4195(2)	61(1)*
C(30)	6001(2)	1873(2)	5079(2)	73(1)*
C(31)	6044(2)	2373(2)	5957(2)	64(1)*
C(32)	5175(2)	2928(2)	5960(2)	62(1)*
C(33)	4270(2)	3006(2)	5076(2)	54(1)*
O(w)	1729(1)	2662(1)	5290(1)	45(1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2g Bond lengths (A)

N(1)-C(2)	1.478(2)	N(1)-C(6)	1.468(3)
N(1)-N(7)	1.418(2)	C(2)-N(3)	1.451(3)
N(3)-C(4)	1.465(3)	N(3)-N(16)	1.418(2)
C(4)-N(5)	1.465(3)	N(5)-C(6)	1.446(3)
N(5)-N(25)	1.408(2)	N(7)-C(8)	1.345(3)
C(8)-O(9)	1.223(2)	C(8)-C(10)	1.497(3)
C(10)-C(11)	1.390(3)	C(10)-C(15)	1.385(2)
C(11)-C(12)	1.383(3)	C(12)-C(13)	1.376(3)
C(13)-C(14)	1.366(4)	C(14)-C(15)	1.379(3)
N(16)-C(17)	1.349(2)	C(17)-O(18)	1.229(2)
C(17)-C(19)	1.490(2)	C(19)-C(20)	1.380(3)
C(19)-C(24)	1.385(3)	C(20)-C(21)	1.390(3)
C(21)-C(22)	1.354(4)	C(22)-C(23)	1.368(4)
C(23)-C(24)	1.379(3)	N(25)-C(26)	1.343(3)
C(26)-O(27)	1.226(3)	C(26)-C(28)	1.494(2)
C(28)-C(29)	1.382(3)	C(28)-C(33)	1.385(3)
C(29)-C(30)	1.375(3)	C(30)-C(31)	1.370(4)
C(31)-C(32)	1.370(4)	C(32)-C(33)	1.384(3)

TABLE 3G Bond angles (deg.)

C(2)-N(1)-C(6)	109.3(1)	C(2)-N(1)-N(7)	109.8(1)
C(6)-N(1)-N(7)	109.1(1)	N(1)-C(2)-N(3)	110.2(2)
C(2)-N(3)-C(4)	110.5(2)	C(2)-N(3)-N(16)	110.7(1)
C(4)-N(3)-N(16)	112.9(1)	N(3)-C(4)-N(5)	116.3(2)
C(4)-N(5)-C(6)	111.0(2)	C(4)-N(5)-N(25)	113.1(1)
C(6)-N(5)-N(25)	112.0(1)	N(1)-C(6)-N(5)	109.9(1)
N(1)-N(7)-C(8)	118.9(1)	N(7)-C(8)-O(9)	123.1(2)
N(7)-C(8)-C(10)	115.2(1)	O(9)-C(8)-C(10)	121.7(2)
C(8)-C(10)-C(11)	119.0(2)	C(8)-C(10)-C(15)	122.9(2)
C(11)-C(10)-C(15)	119.0(2)	C(10)-C(11)-C(12)	119.6(2)
C(11)-C(12)-C(13)	120.9(2)	C(12)-C(13)-C(14)	119.4(2)
C(13)-C(14)-C(15)	120.6(2)	C(10)-C(15)-C(14)	120.4(2)
N(3)-N(16)-C(17)	119.3(1)	N(16)-C(17)-O(18)	123.1(2)
N(16)-C(17)-C(19)	115.2(2)	O(18)-C(17)-C(19)	121.7(2)
C(17)-C(19)-C(20)	118.5(2)	C(17)-C(19)-C(24)	123.1(2)
C(20)-C(19)-C(24)	118.4(2)	C(19)-C(20)-C(21)	120.6(2)
C(20)-C(21)-C(22)	120.4(2)	C(21)-C(22)-C(23)	119.6(2)
C(22)-C(23)-C(24)	120.9(2)	C(19)-C(24)-C(23)	120.1(2)
N(5)-N(25)-C(26)	118.5(2)	N(25)-C(26)-O(27)	123.1(2)
N(25)-C(26)-C(28)	115.2(2)	O(27)-C(26)-C(28)	121.7(2)
C(26)-C(28)-C(29)	118.5(2)	C(26)-C(28)-C(33)	122.6(2)
C(29)-C(28)-C(33)	118.9(2)	C(28)-C(29)-C(30)	120.6(2)
C(29)-C(30)-C(31)	120.4(2)	C(30)-C(31)-C(32)	119.6(2)
C(31)-C(32)-C(33)	120.6(2)	C(28)-C(33)-C(32)	119.9(2)

TABLE 4g Anisotropic temperature factors ($A^2 \times 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	41(1)	41(1)	32(1)	-5(1)	13(1)	-7(1)
C(2)	41(1)	48(1)	43(1)	-3(1)	12(1)	1(1)
N(3)	48(1)	44(1)	31(1)	-2(1)	6(1)	-2(1)
C(4)	53(1)	47(1)	29(1)	3(1)	11(1)	-3(1)
N(5)	40(1)	51(1)	29(1)	-1(1)	9(1)	-2(1)
C(6)	45(1)	47(1)	31(1)	-5(1)	11(1)	0(1)
N(7)	50(1)	51(1)	29(1)	-10(1)	11(1)	-16(1)
C(8)	44(1)	39(1)	35(1)	-3(1)	11(1)	-0(1)
O(9)	67(1)	72(1)	32(1)	-1(1)	8(1)	-23(1)
C(10)	43(1)	38(1)	42(1)	-3(1)	16(1)	2(1)
C(11)	63(1)	45(1)	51(1)	0(1)	21(1)	-2(1)
C(12)	85(2)	47(1)	72(2)	7(1)	38(1)	-4(1)
C(13)	67(2)	50(1)	92(2)	-8(1)	37(1)	-13(1)
C(14)	48(1)	64(2)	78(2)	-8(1)	16(1)	-10(1)
C(15)	46(1)	49(1)	55(1)	-0(1)	16(1)	-1(1)
N(16)	55(1)	34(1)	29(1)	4(1)	9(1)	1(1)
C(17)	48(1)	33(1)	36(1)	2(1)	14(1)	4(1)
O(18)	90(1)	41(1)	43(1)	11(1)	13(1)	-4(1)
C(19)	46(1)	34(1)	39(1)	-2(1)	14(1)	3(1)
C(20)	72(2)	68(2)	53(1)	-2(1)	20(1)	-24(1)
C(21)	63(2)	89(2)	68(2)	-13(1)	8(1)	-29(1)
C(22)	72(2)	72(2)	51(1)	-8(1)	1(1)	3(1)
C(23)	90(2)	53(1)	38(1)	-3(1)	12(1)	-5(1)
C(24)	68(1)	41(1)	38(1)	-3(1)	15(1)	-7(1)
N(25)	40(1)	65(1)	29(1)	-1(1)	12(1)	2(1)
C(26)	47(1)	40(1)	37(1)	1(1)	16(1)	1(1)
O(27)	59(1)	86(1)	39(1)	0(1)	22(1)	16(1)
C(28)	41(1)	46(1)	41(1)	5(1)	18(1)	2(1)
C(29)	63(1)	71(2)	51(1)	2(1)	17(1)	21(1)
C(30)	60(2)	86(2)	68(2)	8(1)	13(1)	27(1)
C(31)	49(1)	78(2)	58(1)	17(1)	4(1)	0(1)
C(32)	49(1)	85(2)	49(1)	-14(1)	10(1)	-10(1)
C(33)	43(1)	66(1)	52(1)	-12(1)	13(1)	2(1)
O(w)	54(1)	48(1)	29(1)	4(1)	10(1)	-2(1)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$$

TABLE 5G Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U
H(2a)	-1110(15)	3390(13)	2150(14)	58(6)
H(2b)	-973(14)	3521(12)	3446(14)	52(5)
H(4a)	434(14)	3857(12)	1526(14)	50(5)
H(4b)	1609(15)	4264(13)	2439(15)	59(6)
H(6a)	-7(14)	2237(11)	1683(13)	41(5)
H(6b)	831(15)	1620(13)	2559(14)	57(6)
H(7)	-1153(15)	1510(13)	2580(14)	58(6)
H(11)	-1027(16)	251(14)	5421(15)	65(6)
H(12)	-2340(17)	-853(15)	5312(16)	80(7)
H(13)	-3864(17)	-1064(15)	3883(16)	82(7)
H(14)	-4079(18)	-32(16)	2510(17)	86(7)
H(15)	-2806(14)	1119(12)	2570(14)	51(5)
H(16)	1090(14)	3811(13)	4435(14)	52(5)
H(20)	3071(16)	6101(15)	5069(16)	73(7)
H(21)	4201(19)	6317(19)	6800(19)	112(9)
H(22)	3912(20)	5455(18)	8167(19)	109(9)
H(23)	2346(20)	4429(18)	7800(19)	108(9)
H(24)	1209(16)	4303(14)	6071(15)	65(6)
H(25)	2205(15)	2740(13)	3974(14)	57(6)
H(29)	5049(18)	1618(15)	3559(16)	87(7)
H(30)	6642(20)	1504(18)	5056(19)	115(9)
H(31)	6643(17)	2304(15)	6556(16)	76(7)
H(32)	5174(18)	3271(15)	6562(16)	82(7)
H(33)	3685(14)	3418(12)	5088(14)	55(5)
H(1w)	1051(20)	2367(16)	5133(18)	99(8)
H(2w)	2092(16)	2584(14)	5904(16)	69(6)

TABLE 1h Atom coordinates ($x10^4$) and temperature factors (A^2x10^3)

atom	x/a	y/b	z/c	U_{eq}
N(1)	6108(1)	2954(1)	4735(1)	51(1)*
N(2)	6239(1)	1619(1)	5002(1)	54(1)*
C(3)	5651(1)	1073(2)	4013(2)	51(1)*
N(4)	5148(1)	1875(1)	2981(1)	48(1)*
C(5)	5367(1)	3204(2)	3479(2)	52(1)*
C(6)	6550(1)	3930(2)	5525(2)	49(1)*
C(7)	7345(1)	3709(2)	6767(2)	48(1)*
C(8)	7600(1)	2536(2)	7426(2)	66(1)*
C(9)	8343(2)	2463(2)	8581(2)	78(1)*
C(10)	8846(1)	3549(2)	9088(2)	74(1)*
C(11)	8594(1)	4713(2)	8451(2)	73(1)*
C(12)	7848(1)	4800(2)	7310(2)	62(1)*
O(6)	6294(1)	5045(1)	5160(1)	63(1)*
N(13)	4207(1)	1595(1)	2424(1)	50(1)*
C(14)	3586(1)	1569(1)	3273(2)	44(1)*
O(14)	3839(1)	1708(1)	4556(1)	53(1)*
C(15)	2579(1)	1378(1)	2557(2)	46(1)*
C(16)	2213(1)	1772(2)	1187(2)	58(1)*
C(17)	1265(1)	1634(2)	614(2)	67(1)*
C(18)	681(1)	1097(2)	1396(2)	67(1)*
C(19)	1047(1)	683(2)	2752(2)	69(1)*
C(20)	1992(1)	822(2)	3334(2)	58(1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2h Bond lengths (A)

N(1)-N(2)	1.410(2)	N(1)-C(5)	1.467(2)
N(1)-C(6)	1.345(2)	N(2)-C(3)	1.273(2)
C(3)-N(4)	1.383(2)	N(4)-C(5)	1.468(2)
N(4)-N(13)	1.393(2)	C(6)-C(7)	1.499(2)
C(6)-O(6)	1.240(2)	C(7)-C(8)	1.384(2)
C(7)-C(12)	1.386(2)	C(8)-C(9)	1.383(3)
C(9)-C(10)	1.373(3)	C(10)-C(11)	1.367(3)
C(11)-C(12)	1.377(2)	N(13)-C(14)	1.353(2)
C(14)-O(14)	1.228(2)	C(14)-C(15)	1.497(2)
C(15)-C(16)	1.384(2)	C(15)-C(20)	1.384(2)
C(16)-C(17)	1.382(2)	C(17)-C(18)	1.377(3)
C(18)-C(19)	1.378(3)	C(19)-C(20)	1.380(2)

TABLE 3h bond angles (deg.)

N(2)-N(1)-C(5)	111.8(1)	N(2)-N(1)-C(6)	126.9(1)
C(5)-N(1)-C(6)	121.2(1)	N(1)-N(2)-C(3)	104.7(1)
N(2)-C(3)-N(4)	116.5(1)	C(3)-N(4)-C(5)	106.2(1)
C(3)-N(4)-N(13)	118.6(1)	C(5)-N(4)-N(13)	116.6(1)
N(1)-C(5)-N(4)	100.2(1)	N(1)-C(6)-C(7)	122.4(1)
N(1)-C(6)-O(6)	117.2(1)	C(7)-C(6)-O(6)	120.3(1)
C(6)-C(7)-C(8)	126.1(1)	C(6)-C(7)-C(12)	115.9(1)
C(8)-C(7)-C(12)	118.0(1)	C(7)-C(8)-C(9)	120.5(2)
C(8)-C(9)-C(10)	120.7(2)	C(9)-C(10)-C(11)	119.2(2)
C(10)-C(11)-C(12)	120.6(2)	C(7)-C(12)-C(11)	121.0(2)
N(4)-N(13)-C(14)	120.2(1)	N(13)-C(14)-O(14)	121.5(1)
N(13)-C(14)-C(15)	116.1(1)	O(14)-C(14)-C(15)	122.4(2)
C(14)-C(15)-C(16)	122.9(2)	C(14)-C(15)-C(20)	117.7(1)
C(16)-C(15)-C(20)	119.4(1)	C(15)-C(16)-C(17)	120.0(2)
C(16)-C(17)-C(18)	120.4(2)	C(17)-C(18)-C(19)	119.6(2)
C(18)-C(19)-C(20)	120.3(2)	C(15)-C(20)-C(19)	120.2(2)

TABLE 4h Anisotropic temperature factors ($A^2 x 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	54(1)	41(1)	49(1)	4(1)	-6(1)	-1(1)
N(2)	58(1)	42(1)	53(1)	0(1)	-5(1)	2(1)
C(3)	53(1)	45(1)	49(1)	-2(1)	-1(1)	3(1)
N(4)	43(1)	54(1)	43(1)	-1(1)	0(1)	-1(1)
C(5)	51(1)	51(1)	49(1)	8(1)	-1(1)	-2(1)
C(6)	51(1)	43(1)	50(1)	2(1)	7(1)	-1(1)
C(7)	47(1)	49(1)	47(1)	-6(1)	8(1)	-0(1)
C(8)	70(1)	54(1)	61(1)	-4(1)	-12(1)	1(1)
C(9)	82(1)	69(1)	68(1)	-7(1)	-18(1)	15(1)
C(10)	59(1)	89(1)	64(1)	-20(1)	-9(1)	11(1)
C(11)	62(1)	76(1)	73(1)	-23(1)	-0(1)	-13(1)
C(12)	63(1)	56(1)	63(1)	-7(1)	6(1)	-7(1)
O(6)	68(1)	45(1)	69(1)	7(1)	-0(1)	1(1)
N(13)	45(1)	65(1)	38(1)	-6(1)	2(1)	-4(1)
C(14)	51(1)	38(1)	43(1)	-0(1)	6(1)	2(1)
O(14)	60(1)	56(1)	40(1)	-3(1)	6(1)	1(1)
C(15)	47(1)	40(1)	49(1)	-2(1)	7(1)	2(1)
C(16)	52(1)	64(1)	55(1)	10(1)	7(1)	4(1)
C(17)	57(1)	77(1)	61(1)	11(1)	-2(1)	5(1)
C(18)	47(1)	69(1)	80(1)	-2(1)	2(1)	-2(1)
C(19)	56(1)	61(1)	73(1)	5(1)	16(1)	-8(1)
C(20)	56(1)	61(1)	55(1)	2(1)	11(1)	-4(1)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2 (h^2 a^{*2} U_{11} + k^2 b^{*2} U_{22} + \ldots + 2hka^*b^* U_{12})$$

TABLE 5h Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U
H(3)	5579(11)	113(17)	3949(16)	64(5)
H(5a)	4799(12)	3668(16)	3724(17)	58(5)
H(20)	2268(12)	489(17)	4292(17)	73(5)
H(5b)	5625(12)	3706(16)	2834(18)	58(5)
H(18)	24(12)	983(17)	1027(18)	73(5)
H(13)	4069(13)	1388(17)	1554(19)	75(5)
H(10)	9377(15)	3482(18)	9933(21)	88(6)
H(16)	2610(12)	2125(17)	644(17)	71(5)
H(17)	1015(14)	1933(18)	-337(19)	83(6)
H(19)	618(14)	349(19)	3291(19)	93(6)
H(8)	7236(15)	1786(18)	7050(20)	77(6)
H(9)	8551(17)	1626(22)	8974(25)	116(8)
H(12)	7638(12)	5615(18)	6849(17)	75(5)
H(11)	8912(15)	5474(22)	8806(21)	101(7)

TABLE 1i Atom coordinates ($x10^4$) and temperature factors (A^2x10^3)

atom	x/a	y/b	z/c	U_{eq}
N(1)	11097(3)	2273(2)	2142(2)	52(1)*
N(2)	9636(3)	2549(2)	3018(2)	54(1)*
C(3)	9000(4)	1595(3)	3681(3)	53(1)*
N(4)	9366(3)	545(2)	3903(2)	48(1)*
C(5)	10910(4)	-267(3)	2758(3)	53(1)*
N(6)	12201(3)	174(2)	1998(2)	51(1)*
N(7)	11863(3)	509(2)	950(2)	51(1)*
C(8)	11032(4)	1795(3)	1051(3)	59(1)*
C(9)	12304(4)	1346(3)	2420(2)	54(1)*
C(10)	9200(4)	3669(3)	3526(3)	68(2)*
O(11)	8270(3)	3899(2)	4509(2)	98(1)*
C(12)	9840(4)	4637(3)	2861(3)	63(2)*
C(13)	9466(6)	4974(4)	1895(3)	104(2)*
C(14)	9976(7)	5923(4)	1303(4)	131(3)*
C(15)	10846(6)	6522(3)	1698(4)	110(3)*
C(16)	11237(5)	6174(4)	2661(4)	96(2)*
C(17)	10718(4)	5242(3)	3259(3)	81(2)*
N(18)	8160(3)	-67(2)	3119(2)	54(1)*
C(19)	7832(4)	-724(3)	4048(3)	53(1)*
O(20)	8428(3)	-703(2)	4820(2)	71(1)*
C(21)	6729(4)	-1494(3)	4052(2)	50(1)*
C(22)	6882(4)	-2530(3)	4620(3)	72(2)*
C(23)	5929(5)	-3304(3)	4617(4)	85(2)*
C(24)	4812(4)	-3040(4)	4066(4)	83(2)*
C(25)	4633(4)	-2002(4)	3524(4)	90(2)*
C(26)	5593(4)	-1230(3)	3504(3)	73(2)*
C(27)	12488(4)	-112(3)	-78(3)	54(1)*
O(28)	12137(3)	373(2)	-909(2)	77(1)*
C(29)	13515(4)	-1349(3)	-223(3)	61(1)*
C(30)	14510(4)	-1776(3)	422(3)	75(2)*
C(31)	15473(5)	-2936(4)	176(4)	107(2)*
C(32)	15453(6)	-3660(4)	-716(4)	124(3)*
C(33)	14485(5)	-3232(3)	-1362(4)	117(2)*
C(34)	13526(4)	-2088(3)	-1134(3)	83(2)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2i Bond lengths (A)

N(1)-N(2)	1.412(3)	N(1)-C(8)	1.458(4)
N(1)-C(9)	1.464(4)	N(2)-C(3)	1.482(4)
N(2)-C(10)	1.345(4)	C(3)-N(4)	1.445(4)
N(4)-C(5)	1.466(4)	N(4)-N(18)	1.406(4)
C(5)-N(6)	1.464(4)	N(6)-N(7)	1.426(4)
N(6)-C(9)	1.473(4)	N(7)-C(8)	1.481(4)
N(7)-C(27)	1.337(3)	C(10)-O(11)	1.232(4)
C(10)-C(12)	1.498(5)	C(12)-C(13)	1.347(6)
C(12)-C(17)	1.364(6)	C(13)-C(14)	1.396(6)
C(14)-C(15)	1.352(8)	C(15)-C(16)	1.354(7)
C(16)-C(17)	1.386(6)	N(18)-C(19)	1.351(4)
C(19)-O(20)	1.219(4)	C(19)-C(21)	1.490(5)
C(21)-C(22)	1.370(5)	C(21)-C(26)	1.374(5)
C(22)-C(23)	1.386(6)	C(23)-C(24)	1.361(7)
C(24)-C(25)	1.357(6)	C(25)-C(26)	1.383(6)
C(27)-O(28)	1.239(4)	C(27)-C(29)	1.487(4)
C(29)-C(30)	1.379(5)	C(29)-C(34)	1.391(5)
C(30)-C(31)	1.392(5)	C(31)-C(32)	1.373(8)
C(32)-C(33)	1.361(8)	C(33)-C(34)	1.375(5)

TABLE 3i Bond angles (deg.)

N(2)-N(1)-C(8)	113.8(3)	N(2)-N(1)-C(9)	114.0(2)
C(8)-N(1)-C(9)	103.4(2)	N(1)-N(2)-C(3)	121.3(2)
N(1)-N(2)-C(10)	115.0(3)	C(3)-N(2)-C(10)	118.3(2)
N(2)-C(3)-N(4)	108.5(2)	C(3)-N(4)-C(5)	114.6(3)
C(3)-N(4)-N(18)	113.7(2)	C(5)-N(4)-N(18)	112.6(2)
N(4)-C(5)-N(6)	113.1(2)	C(5)-N(6)-N(7)	109.7(3)
C(5)-N(6)-C(9)	113.2(2)	N(7)-N(6)-C(9)	99.4(2)
N(6)-N(7)-C(8)	110.6(2)	N(6)-N(7)-C(27)	126.7(2)
C(8)-N(7)-C(27)	121.0(2)	N(1)-C(8)-N(7)	104.9(2)
N(1)-C(9)-N(6)	107.4(3)	N(2)-C(10)-O(11)	120.7(3)
N(2)-C(10)-C(12)	118.6(3)	O(11)-C(10)-C(12)	120.7(3)
C(10)-C(12)-C(13)	120.0(4)	C(10)-C(12)-C(17)	120.9(3)
C(13)-C(12)-C(17)	119.0(4)	C(12)-C(13)-C(14)	120.7(5)
C(13)-C(14)-C(15)	120.2(5)	C(14)-C(15)-C(16)	119.0(4)
C(15)-C(16)-C(17)	120.9(5)	C(12)-C(17)-C(16)	120.1(4)
N(4)-N(18)-C(19)	121.9(3)	N(18)-C(19)-O(20)	122.0(3)
N(18)-C(19)-C(21)	115.6(3)	O(20)-C(19)-C(21)	122.4(3)
C(19)-C(21)-C(22)	117.7(3)	C(19)-C(21)-C(26)	123.6(3)
C(22)-C(21)-C(26)	118.6(4)	C(21)-C(22)-C(23)	120.3(4)
C(22)-C(23)-C(24)	120.6(4)	C(23)-C(24)-C(25)	119.2(4)
C(24)-C(25)-C(26)	120.8(4)	C(21)-C(26)-C(25)	120.4(3)
N(7)-C(27)-O(28)	117.1(3)	N(7)-C(27)-C(29)	122.0(3)
O(28)-C(27)-C(29)	120.9(3)	C(27)-C(29)-C(30)	125.0(3)
C(27)-C(29)-C(34)	116.2(3)	C(30)-C(29)-C(34)	118.7(3)
C(29)-C(30)-C(31)	120.1(4)	C(30)-C(31)-C(32)	120.5(5)
C(31)-C(32)-C(33)	119.4(4)	C(32)-C(33)-C(34)	121.0(4)
C(29)-C(34)-C(33)	120.3(4)		

TABLE 4i Anisotropic temperature factors ($A^2 \times 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	64(2)	49(2)	39(1)	8(1)	-10(1)	-16(1)
N(2)	66(2)	43(1)	45(2)	2(1)	-2(1)	-17(1)
C(3)	67(2)	50(2)	43(2)	4(1)	-14(2)	-19(2)
N(4)	63(2)	44(1)	42(1)	4(1)	-15(1)	-22(1)
C(5)	73(2)	46(2)	43(2)	9(1)	-19(2)	-17(2)
N(6)	66(2)	54(2)	36(1)	12(1)	-16(1)	-18(1)
N(7)	66(2)	51(2)	32(1)	4(1)	-14(1)	-5(1)
C(8)	70(2)	62(2)	44(2)	13(2)	-18(2)	-15(2)
C(9)	66(2)	63(2)	40(2)	8(2)	-16(2)	-24(2)
C(10)	82(2)	55(2)	59(2)	-0(2)	-5(2)	-22(2)
O(11)	126(2)	69(2)	74(2)	-13(1)	18(2)	-38(2)
C(12)	86(3)	43(2)	54(2)	-1(2)	-8(2)	-19(2)
C(13)	165(4)	88(3)	92(3)	30(2)	-61(3)	-64(3)
C(14)	222(6)	92(3)	104(4)	44(3)	-68(4)	-65(4)
C(15)	182(5)	58(3)	80(3)	9(2)	-7(3)	-53(3)
C(16)	113(3)	81(3)	98(3)	0(3)	-12(3)	-56(3)
C(17)	95(3)	76(3)	75(3)	8(2)	-20(2)	-36(2)
N(18)	77(2)	60(2)	38(1)	15(1)	-23(1)	-37(1)
C(19)	68(2)	56(2)	41(2)	12(2)	-17(2)	-23(2)
O(20)	95(2)	95(2)	45(1)	26(1)	-34(1)	-47(1)
C(21)	58(2)	53(2)	39(2)	8(1)	-10(2)	-21(2)
C(22)	81(2)	71(2)	74(2)	28(2)	-29(2)	-31(2)
C(23)	89(3)	61(2)	105(3)	25(2)	-20(3)	-31(2)
C(24)	73(3)	88(3)	94(3)	11(2)	-13(2)	-45(2)
C(25)	73(3)	117(3)	99(3)	30(3)	-39(2)	-46(3)
C(26)	70(2)	79(3)	82(3)	31(2)	-30(2)	-32(2)
C(27)	61(2)	64(2)	42(2)	6(2)	-15(2)	-27(2)
O(28)	103(2)	90(2)	39(1)	5(1)	-26(1)	-20(2)
C(29)	63(2)	61(2)	50(2)	3(2)	1(2)	-21(2)
C(30)	71(2)	72(2)	67(2)	7(2)	-3(2)	-9(2)
C(31)	77(3)	89(3)	120(4)	26(3)	6(3)	2(3)
C(32)	119(4)	60(3)	141(5)	4(3)	22(4)	-1(3)
C(33)	126(4)	72(3)	125(4)	-29(3)	8(3)	-26(3)
C(34)	87(3)	75(3)	78(3)	-16(2)	-2(2)	-31(2)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2 (h^2 a^{*2} U_{11} + k^2 b^{*2} U_{22} + \ldots + 2hka^*b^* U_{12})$$

TABLE 5i Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U
H(13)	8840	4559	1612	114
H(14)	9708	6150	612	143
H(15)	11184	7184	1301	120
H(16)	11880	6579	2935	105
H(17)	10977	5021	3954	88
H(22)	7654	-2720	5022	78
H(23)	6057	-4034	5008	93
H(24)	4159	-3581	4061	91
H(25)	3831	-1801	3149	98
H(26)	5465	-506	3105	79
H(30)	14537	-1273	1043	82
H(31)	16158	-3231	631	116
H(32)	16114	-4461	-882	135
H(33)	14472	-3737	-1987	130
H(34)	12862	-1798	-1605	90
H(3a)	9438(30)	1414(21)	4335(22)	58(8)
H(3b)	7883(31)	1900(22)	3991(22)	66(8)
H(5a)	11154(26)	-417(19)	3482(19)	43(7)
H(5b)	10914(29)	-1065(21)	2397(22)	61(8)
H(8a)	10010(29)	1886(21)	1026(21)	55(8)
H(8b)	11657(33)	2148(24)	381(24)	77(9)
H(9a)	13409(30)	1445(23)	1956(23)	56(8)
H(9b)	12158(25)	1381(18)	3212(18)	37(6)
H(18)	7992(27)	-225(20)	2498(21)	52(7)

TABLE 1j Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
N(1)	2240(2)	2848(1)	6836(1)	33(1)*
C(2)	406(2)	2943(2)	6248(1)	33(1)*
C(3)	350(2)	4180(2)	5468(1)	34(1)*
N(4)	2116(2)	5164(2)	5411(1)	37(1)*
C(5)	3981(3)	5024(2)	6030(1)	42(1)*
C(6)	4042(3)	3867(2)	6747(1)	39(1)*
N(7)	1999(3)	1598(2)	7508(1)	45(1)*
N(8)	81(3)	995(2)	7315(1)	52(1)*
N(9)	-971(3)	1806(2)	6536(1)	48(1)*
N(10)	-1098(3)	4629(2)	4763(1)	46(1)*
N(11)	-164(3)	5900(2)	4260(1)	52(1)*
N(12)	1757(3)	6242(2)	4635(1)	50(1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2j Bond lengths (A)

N(1)-C(2)	1.351(2)	N(1)-C(6)	1.391(2)
N(1)-N(7)	1.349(2)	C(2)-C(3)	1.426(2)
C(2)-N(9)	1.313(2)	C(3)-N(4)	1.352(2)
C(3)-N(10)	1.315(2)	N(4)-C(5)	1.392(2)
N(4)-N(12)	1.348(2)	C(5)-C(6)	1.323(2)
N(7)-N(8)	1.298(2)	N(8)-N(9)	1.359(2)
N(10)-N(11)	1.356(2)	N(11)-N(12)	1.299(2)

TABLE 3j Bond angles (deg.)

C(2)-N(1)-C(6)	125.2(1)	C(2)-N(1)-N(7)	107.8(1)
C(6)-N(1)-N(7)	126.9(1)	N(1)-C(2)-C(3)	116.6(1)
N(1)-C(2)-N(9)	109.6(1)	C(3)-C(2)-N(9)	133.8(1)
C(2)-C(3)-N(4)	116.7(1)	C(2)-C(3)-N(10)	133.9(1)
N(4)-C(3)-N(10)	109.4(1)	C(3)-N(4)-C(5)	125.0(1)
C(3)-N(4)-N(12)	107.8(1)	C(5)-N(4)-N(12)	127.2(1)
N(4)-C(5)-C(6)	118.3(1)	N(1)-C(6)-C(5)	118.1(1)
N(1)-N(7)-N(8)	105.8(1)	N(7)-N(8)-N(9)	112.1(1)
C(2)-N(9)-N(8)	104.6(1)	C(3)-N(10)-N(11)	104.8(1)
N(10)-N(11)-N(12)	112.1(1)	N(4)-N(12)-N(11)	105.8(1)

TABLE 4j Anisotropic temperature factors ($A^2 x 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	35(1)	33(1)	32(1)	0(1)	-1(1)	1(1)
C(2)	32(1)	32(1)	34(1)	-4(1)	-0(1)	-0(1)
C(3)	37(1)	34(1)	32(1)	-6(1)	-0(1)	1(1)
N(4)	44(1)	32(1)	33(1)	0(1)	2(1)	-3(1)
C(5)	35(1)	43(1)	47(1)	-3(1)	1(1)	-7(1)
C(6)	32(1)	43(1)	43(1)	-3(1)	-2(1)	1(1)
N(7)	52(1)	41(1)	42(1)	7(1)	-0(1)	2(1)
N(8)	56(1)	46(1)	54(1)	14(1)	3(1)	-9(1)
N(9)	44(1)	45(1)	54(1)	5(1)	0(1)	-12(1)
N(10)	53(1)	46(1)	39(1)	-2(1)	-11(1)	7(1)
N(11)	71(1)	45(1)	40(1)	3(1)	-7(1)	7(1)
N(12)	69(1)	38(1)	41(1)	5(1)	0(1)	1(1)
H(5)	47	51	62	2	1	-17
H(6)	37	57	53	-1	-10	0

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2 a^{*2} U_{11} + k^2 b^{*2} U_{22} + \ldots + 2hka^*b^* U_{12})$$

TABLE 5j Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x 10^3$)

atom	x/a	y/b	z/c	U
H(5)	4984(31)	5828(27)	5964(14)	53*
H(6)	5194(29)	3690(21)	7215(14)	49*

Table 1k Atomic coordinates and temperature factors $(Å)^2$*

Atom	x/a	y/b	z/c	U_{eq}
Br1	0.1041(2)	0.0615(2)	1.1220(1)	4.3(1)
Br2	0.3816(2)	0.3325(2)	1.1286(1)	4.7(1)
N1	0.3786(12)	0.1538(11)	0.9362(7)	2.7(3)
N2	0.2374(15)	0.3193(14)	0.8724(8)	4.2(4)
O1	0.1171(13)	0.2899(14)	0.8356(8)	5.9(4)
O2	0.2631(13)	0.4725(12)	0.8598(7)	5.4(4)
C2	0.3116(15)	-.0003(15)	0.9822(8)	2.9(4)
C6	0.5093(15)	0.1914(14)	0.9879(8)	3.0(4)
Br1'	0.3341(2)	0.8776(2)	0.6236(1)	4.9(1)
Br2'	0.0599(2)	0.6119(2)	0.6114(1)	4.6(1)
N1'	0.1563(12)	0.8795(12)	0.4304(7)	3.0(3)
N2'	0.3232(15)	0.7538(14)	0.3587(8)	4.6(4)
O1'	0.4685(13)	0.7889(15)	0.3337(8)	5.9(4)
O2'	0.2944(15)	0.6252(14)	0.3252(8)	6.3(5)
C2'	0.1942(16)	1.0104(16)	0.4863(8)	3.2(4)
C6'	-.0015(16)	0.8172(15)	0.4772(9)	3.2(4)

Hydrogen Coordinates

H2	0.251	-.014	0.923	3.5
H6	0.552	0.278	0.929	3.6
H2'	0.282	1.056	0.429	4.0
H6'	-.016	0.761	0.415	3.8

Coordinates are given for both half-molecules in the asymmetric unit. The remaining half of each molecule is generated by crystallographic symmetry (1-x, -y, 2-z for the unprimed half-molecule; -x, 2-y, 1-z for the primed one).

*
Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor.

Table 2k Bond lengths (Å)

	Molecule 1	Molecule 2(')
Br1-C2	1.980(9)	1.984(10)
Br2-C6	1.984(10)	1.971(10)
N1-C2	1.437(16)	1.443(18)
N1-C6	1.410(17)	1.429(16)
N1-N2	1.441(1)	1.412(11)
N2-O1	1.201(17)	1.198(17)
N2-O2	1.233(16)	1.234(16)
C2-C6A	1.525(12)	1.505(13)

Table 3k Bond angles (deg.)

	Molecule 1	Molecule 2(')
C2-N1-C6	123.6(8)	122.4(8)
C2-N1-N2	113.1(9)	115.3(9)
C6-N1-N2	117.3(9)	117.4(10)
N1-N2-O1	117.2(10)	117.7(11)
N1-N2-O2	113.9(11)	113.9(10)
O1-N2-O2	128.8(9)	128.3(9)
Br1-C2-N1	113.9(7)	113.6(7)
Br1-C2-C6A	108.9(6)	109.0(7)
N1-C2-C6A	109.2(9)	110.6(10)
Br2-C6-N1	114.6(6)	113.3(7)
Br2-C6-C2A	108.7(7)	108.4(6)
N1-C6-C2A	111.2(10)	111.2(10)

Table 4k Torsion angles (deg.)

	Molecule 1	Molecule 2
N1-C2-C6A-N1A	40.3	40.6
C2-C6A-N1A-C2A	-47.1	-46.2
C6A-N1A-C2A-C6	46.3	45.9
Br1-N1A-C2A-Br2A	148.4	149.8
C2-N1-N2-O1	-21.3	-12.9
C2-N1-N2-O2	161.2	170.4
C6-N1-N2-O1	-175.0	-168.9
C6-N1-N2-O2	7.5	14.4

TABLE 1| Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
N(1)	-328(3)	629(4)	2225(1)	48(1)*
C(2)	-1052(4)	874(5)	1789(2)	47(1)*
N(3)	-526(3)	1064(4)	1274(1)	47(1)*
C(4)	-390(4)	2381(4)	1019(2)	46(1)*
N(5)	-1267(3)	2767(4)	662(1)	44(1)*
C(6)	-1350(4)	2312(5)	114(2)	45(1)*
N(7)	-1120(3)	3356(4)	-283(1)	59(1)*
C(10)	186(4)	1667(5)	2468(2)	46(1)*
O(11)	65(3)	2865(3)	2321(1)	57(1)*
C(12)	877(5)	1271(6)	2923(2)	72(2)*
N(13)	-80(3)	-76(4)	1043(2)	55(1)*
O(14)	356(3)	67(4)	608(1)	71(1)*
O(15)	-161(3)	-1172(4)	1286(2)	71(1)*
N(16)	-2058(3)	3585(4)	855(2)	51(1)*
O(17)	-2714(3)	4034(4)	538(1)	69(1)*
O(18)	-2063(3)	3821(4)	1337(1)	70(1)*
N(19)	-57(4)	3642(4)	-382(2)	70(2)*
O(20)	609(3)	3127(4)	-84(2)	82(1)*
O(21)	144(4)	4396(4)	-756(2)	112(2)*
C(8)	-1922(5)	3880(6)	-627(2)	104(2)*
O(9)	-1702(5)	3723(6)	-1177(2)	84(2)*
C(22)	-2246(6)	2481(7)	-1280(3)	53(2)*
O(23)	-2774(6)	1916(7)	-929(2)	78(2)*
C(24)	-2295(8)	2064(10)	-1857(3)	89(3)*
O(9a)	-2113(9)	2692(11)	-1005(3)	103(3)*
C(22a)	-2590(8)	2593(3)	-1518(3)	106(3)*
O(23a)	-2521(16)	3792(4)	-1674(4)	265(3)*
C(24a)	-2199(12)	1383(4)	-1840(5)	75(3)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2| Bond lengths (A)

N(1)-C(2)	1.434(6)	N(1)-C(10)	1.338(6)
C(2)-N(3)	1.460(6)	N(3)-C(4)	1.438(6)
N(3)-N(13)	1.364(6)	C(4)-N(5)	1.458(6)
N(5)-C(6)	1.447(5)	N(5)-N(16)	1.351(5)
C(6)-N(7)	1.448(6)	N(7)-N(19)	1.370(7)
N(7)-C(8)	1.413(7)	C(10)-O(11)	1.228(5)
C(10)-C(12)	1.479(7)	N(13)-O(14)	1.225(6)
N(13)-O(15)	1.228(6)	N(16)-O(17)	1.217(5)
N(16)-O(18)	1.231(5)	N(19)-O(20)	1.221(6)
N(19)-O(21)	1.216(6)	C(8)-O(9)	1.413(7)
C(8)-O(9a)	1.509(11)	O(9)-C(22)	1.404(9)
C(22)-O(23)	1.226(10)	C(22)-C(24)	1.504(10)
O(9a)-C(22a)	1.421(12)	C(22a)-O(23a)	1.229(6)
C(22a)-C(24a)	1.504(10)		

TABLE 3| Bond angles (deg.)

C(2)-N(1)-C(10)	121.5(4)	N(1)-C(2)-N(3)	114.5(4)
C(2)-N(3)-C(4)	123.9(4)	C(2)-N(3)-N(13)	117.0(4)
C(4)-N(3)-N(13)	118.9(4)	N(3)-C(4)-N(5)	114.4(4)
C(4)-N(5)-C(6)	123.9(3)	C(4)-N(5)-N(16)	118.3(3)
C(6)-N(5)-N(16)	117.8(3)	N(5)-C(6)-N(7)	115.2(4)
C(6)-N(7)-N(19)	117.2(4)	C(6)-N(7)-C(8)	122.2(4)
N(19)-N(7)-C(8)	119.6(4)	N(1)-C(10)-O(11)	121.1(4)
N(1)-C(10)-C(12)	115.7(4)	O(11)-C(10)-C(12)	123.3(4)
N(3)-N(13)-O(14)	117.7(4)	N(3)-N(13)-O(15)	117.2(4)
O(14)-N(13)-O(15)	125.1(4)	N(5)-N(16)-O(17)	117.5(4)
N(5)-N(16)-O(18)	117.7(4)	O(17)-N(16)-O(18)	124.8(4)
N(7)-N(19)-O(20)	117.3(4)	N(7)-N(19)-O(21)	117.4(5)
O(20)-N(19)-O(21)	125.3(5)	N(7)-C(8)-O(9)	115.0(5)
N(7)-C(8)-O(9a)	102.7(6)	O(9)-C(22)-O(23)	120.6(7)
C(8)-O(9)-C(22)	100.3(5)	O(23)-C(22)-C(24)	123.2(7)
O(9)-C(22)-C(24)	115.3(7)	C(8)-O(9a)-C(22a)	133.2(8)
O(9a)-C(22a)-O(23a)	101.3(8)	O(9a)-C(22a)-C(24a)	113.9(8)
O(23a)-C(22a)-C(24a)	123.0(9)		

TABLE 4| Anisotropic temperature factors ($A^2 \times 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	66(2)	34(2)	44(2)	1(2)	-9(2)	-2(2)
C(2)	47(3)	44(3)	51(2)	-10(2)	6(2)	-4(2)
N(3)	65(2)	40(2)	37(2)	-11(2)	-1(2)	16(2)
C(4)	48(2)	44(3)	44(2)	-10(2)	-1(2)	6(2)
N(5)	51(2)	44(2)	36(2)	-6(2)	-3(2)	16(2)
C(6)	54(3)	42(2)	40(2)	-4(2)	-5(2)	3(2)
N(7)	80(3)	55(2)	43(2)	6(2)	-3(2)	13(2)
C(10)	62(3)	42(2)	36(2)	-5(2)	1(2)	6(2)
O(11)	81(2)	37(2)	54(2)	-1(2)	-13(2)	-5(2)
C(12)	94(4)	61(3)	62(3)	-2(3)	-20(3)	7(3)
N(13)	65(2)	50(2)	50(2)	-11(2)	-10(2)	17(2)
O(14)	98(3)	66(2)	50(2)	-12(2)	11(2)	29(2)
O(15)	93(3)	43(2)	71(2)	-2(2)	-15(2)	11(2)
N(16)	50(2)	53(2)	48(2)	-6(2)	3(2)	16(2)
O(17)	50(2)	82(2)	64(2)	-6(2)	-10(2)	34(2)
O(18)	73(2)	87(2)	50(2)	-18(2)	5(2)	28(2)
N(19)	109(3)	50(3)	51(2)	-11(2)	22(3)	-1(3)
O(20)	74(2)	100(3)	71(2)	-12(2)	7(2)	-16(2)
O(21)	178(4)	68(2)	91(3)	15(2)	57(3)	-6(3)
C(8)	133(4)	115(4)	44(3)	7(3)	1(3)	78(4)
O(9)	105(4)	72(4)	74(4)	41(3)	-3(4)	-13(3)
C(22)	23(2)	31(3)	104(5)	-15(1)	-24(4)	-14(3)
O(23)	89(4)	72(4)	72(4)	-17(3)	-11(4)	-0(3)
C(24)	73(5)	98(5)	98(5)	37(5)	21(5)	74(5)
O(9a)	107(5)	160(6)	41(4)	-46(4)	-34(4)	49(5)
C(22a)	40(5)	49(5)	223(6)	-11(6)	-28(6)	15(5)
O(23a)	643(6)	63(5)	84(5)	4(4)	-147(6)	27(6)
C(24a)	83(6)	87(6)	54(5)	4(5)	-17(5)	25(5)

The anisotropic temperature factor exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$

TABLE 5| Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
H(1)	-208	-299	2344	45
H(2a)	-1530	98	1762	45
H(2b)	-1461	1690	1867	45
H(4a)	-334	3072	1292	39
H(4b)	265	2356	816	39
H(6a)	-853	1565	64	48
H(6b)	-2072	1989	57	48
H(12a)	1240	421	2843	70
H(12b)	440	1152	3235	70
H(12c)	1401	1981	2987	70
H(8a)	-2611	3489	-541	112
H(8b)	-1936	4855	-562	112
H(8a')	-2536	4182	-426	112
H(8b')	-1635	4641	-827	112
H(24a)	-2681	1212	-1898	91
H(24b)	-1567	1947	-1979	91
H(24c)	-2641	2771	-2063	91
H(24x)	-1967	625	-1622	90
H(24y)	-1681	1541	-2118	90
H(24z)	-2886	1171	-1996	90

TABLE 1m Atom coordinates ($\times 10^4$) and temperature factors ($A^2 \times 10^3$)

atom	x/a	y/b	z/c	U_{eq}
N(1)	2367(3)	1369(1)	2528(2)	47(1)*
C(2)	3136(4)	1823(1)	3493(2)	46(1)*
N(3)	3250(3)	2518(1)	3113(2)	40(1)*
C(4)	4877(3)	2793(1)	2614(2)	38(1)*
N(5)	5857(2)	3312(1)	3369(2)	35(1)*
C(6)	5753(3)	4011(1)	3020(2)	40(1)*
N(7)	7499(3)	4282(1)	2677(2)	44(1)*
C(8)	8577(4)	4790(1)	3417(3)	49(1)*
N(9)	8070(3)	5462(1)	3053(2)	49(1)*
C(10)	3090(3)	1118(1)	1665(2)	45(1)*
O(11)	4990(3)	1321(1)	1586(2)	61(1)*
C(12)	2499(4)	587(2)	822(3)	65(1)*
N(13)	1660(3)	2893(1)	3006(2)	46(1)*
O(14)	213(2)	2597(1)	3165(2)	62(1)*
O(15)	1801(3)	3485(1)	2774(2)	57(1)*
N(16)	6806(3)	3149(1)	4483(2)	39(1)*
O(17)	6733(3)	2566(1)	4831(2)	51(1)*
O(18)	7665(2)	3599(1)	5062(2)	51(1)*
N(19)	7955(3)	4141(1)	1500(2)	47(1)*
O(20)	7046(3)	3710(1)	911(2)	65(1)*
O(21)	9234(3)	4467(1)	1129(2)	61(1)*
C(22)	6701(4)	5795(1)	3540(3)	49(1)*
O(23)	5761(3)	5529(1)	4291(2)	66(1)*
C(24)	6387(5)	6502(2)	3095(3)	64(1)*
N(1')	7453(3)	1470(1)	-289(2)	51(1)*
C(2')	8384(4)	2090(2)	1(2)	49(1)*
N(3')	8202(3)	2572(1)	-1034(2)	44(1)*
C(4')	9676(3)	2727(1)	-1802(2)	40(1)*
N(5')	10597(3)	3359(1)	-1514(2)	42(1)*
C(6')	10515(3)	3922(1)	-2365(2)	40(1)*
N(7')	12199(3)	4029(1)	-2985(2)	44(1)*
C(8')	13446(3)	4592(1)	-2757(3)	49(1)*
N(9')	13180(3)	5117(1)	-3687(3)	50(1)*
C(10')	8070(4)	1027(1)	-1090(3)	48(1)*
O(11')	9411(3)	1170(1)	-1669(2)	59(1)*
C(12')	7072(4)	378(2)	-1253(4)	65(1)*
N(13')	6530(3)	2871(1)	-1346(2)	48(1)*
O(14')	5241(3)	2674(1)	-782(2)	66(1)*
O(15')	6439(3)	3299(1)	-2162(2)	68(1)*
N(16')	11732(3)	3404(1)	-429(2)	44(1)*
O(17')	11814(3)	2915(1)	272(2)	57(1)*
O(18')	12641(3)	3920(1)	-228(2)	56(1)*
N(19')	12561(3)	2591(1)	-3902(2)	46(1)*
O(20')	11627(3)	3085(1)	-4030(2)	58(1)*
O(21')	13819(3)	3750(1)	-4527(2)	63(1)*
C(22')	11925(4)	5566(1)	-2652(3)	55(1)*
O(23')	10748(3)	5536(1)	-2826(3)	81(1)*
C(24')	11690(4)	6101(2)	-4640(4)	65(1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2m Bond lengths (A)

N(1)-C(2)	1.441(3)	N(1)-C(10)	1.341(4)
C(2)-N(3)	1.451(3)	N(3)-C(4)	1.454(3)
N(3)-N(13)	1.369(3)	C(4)-N(5)	1.452(3)
N(5)-C(6)	1.445(3)	N(5)-N(16)	1.354(2)
C(6)-N(7)	1.458(3)	N(7)-C(8)	1.461(3)
N(7)-N(19)	1.364(3)	C(8)-N(9)	1.436(4)
N(9)-C(22)	1.345(4)	C(10)-O(11)	1.239(3)
C(10)-C(12)	1.494(4)	N(13)-O(14)	1.230(3)
N(13)-O(15)	1.216(3)	N(16)-O(17)	1.225(3)
N(16)-O(18)	1.220(3)	N(19)-O(20)	1.218(3)
N(19)-O(21)	1.232(3)	C(22)-O(23)	1.229(4)
C(22)-C(24)	1.500(4)	N(1')-C(2')	1.429(4)
N(1')-C(10')	1.341(4)	C(2')-N(3')	1.463(4)
N(3')-C(4')	1.448(3)	N(3')-N(13')	1.358(3)
C(4')-N(5')	1.447(3)	N(5')-C(6')	1.444(3)
N(5')-N(16')	1.358(3)	C(6')-N(7')	1.465(3)
N(7')-C(8')	1.446(3)	N(7')-N(19')	1.361(3)
C(8')-N(9')	1.444(4)	N(9')-C(22')	1.334(4)
C(10')-O(11')	1.240(4)	C(10')-C(12')	1.487(4)
N(13')-O(14')	1.229(3)	N(13')-O(15')	1.220(3)
N(16')-O(17')	1.230(3)	N(16')-O(18')	1.222(3)
N(19')-O(20')	1.216(3)	N(19')-O(21')	1.229(3)
C(22')-O(23')	1.241(4)	C(22')-C(24')	1.499(5)

TABLE 3m Bond angles (deg.)

C(2)-N(1)-C(10)	121.9(2)	N(1)-C(2)-N(3)	115.5(2)
C(2)-N(3)-C(4)	122.6(2)	C(2)-N(3)-N(13)	118.3(2)
C(4)-N(3)-N(13)	118.1(2)	N(3)-C(4)-N(5)	115.7(2)
C(4)-N(5)-C(6)	122.5(2)	C(4)-N(5)-N(16)	119.4(2)
C(6)-N(5)-N(16)	117.9(2)	N(5)-C(6)-N(7)	113.7(2)
C(6)-N(7)-C(8)	123.3(2)	C(6)-N(7)-N(19)	117.3(2)
C(8)-N(7)-N(19)	118.3(2)	N(7)-C(8)-N(9)	113.4(2)
C(8)-N(9)-C(22)	122.4(2)	N(1)-C(10)-O(11)	121.0(2)
N(1)-C(10)-C(12)	116.5(2)	O(11)-C(10)-C(12)	122.5(3)
N(3)-N(13)-O(14)	116.6(2)	N(3)-N(13)-O(15)	117.5(2)
O(14)-N(13)-O(15)	125.9(2)	N(5)-N(16)-O(17)	117.4(2)
N(5)-N(16)-O(18)	116.8(2)	O(17)-N(16)-O(18)	125.9(2)
N(7)-N(19)-O(20)	116.8(2)	N(7)-N(19)-O(21)	117.0(2)
O(20)-N(19)-O(21)	126.2(2)	N(9)-C(22)-O(23)	121.4(3)
N(9)-C(22)-C(24)	115.9(3)	O(23)-C(22)-C(24)	122.7(3)
C(2')-N(1')-C(10')	121.9(2)	N(1')-C(2')-N(3')	113.9(2)
C(2')-N(3')-C(4')	123.9(2)	C(2')-N(3')-N(13')	118.7(2)
C(4')-N(3')-N(13')	117.3(2)	N(3')-C(4')-N(5')	114.6(2)
C(4')-N(5')-C(6')	124.0(2)	C(4')-N(5')-N(16')	117.9(2)
C(6')-N(5')-N(16')	117.8(2)	N(5')-C(6')-N(7')	114.7(2)
C(6')-N(7')-C(8')	124.9(2)	C(6')-N(7')-N(19')	117.8(2)
C(8')-N(7')-N(19')	117.2(2)	N(7')-C(8')-N(9')	114.5(2)
C(8')-N(9')-C(22')	121.0(3)	N(1')-C(10')-O(11')	119.9(3)
N(1')-C(10')-C(12')	117.4(3)	O(11')-C(10')-C(12')	122.7(3)
N(3')-N(13')-O(14')	116.3(2)	N(3')-N(13')-O(15')	117.5(2)
O(14')-N(13')-O(15')	126.2(2)	N(5')-N(16')-O(17')	116.6(2)
N(5')-N(16')-O(18')	118.1(2)	O(17')-N(16')-O(18')	125.3(2)
N(7')-N(19')-O(20')	117.9(2)	N(7')-N(19')-O(21')	115.9(2)
O(20')-N(19')-O(21')	126.2(2)	N(9')-C(22')-O(23')	120.4(3)
N(9')-C(22')-C(24')	117.0(3)	O(23')-C(22')-C(24')	122.6(3)

TABLE 4m Anisotropic temperature factors ($A^2 \times 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	39(1)	47(1)	57(1)	-7(1)	16(1)	-14(1)
C(2)	48(1)	48(1)	42(1)	3(1)	10(1)	-8(1)
N(3)	32(1)	41(1)	49(1)	-1(1)	9(1)	-5(1)
C(4)	36(1)	40(1)	39(1)	-3(1)	10(1)	-6(1)
N(5)	35(1)	34(1)	36(1)	1(1)	3(1)	-3(1)
C(6)	38(1)	37(1)	45(1)	1(1)	11(1)	-3(1)
N(7)	50(1)	40(1)	44(1)	-2(1)	15(1)	-11(1)
C(8)	47(1)	45(1)	55(1)	-2(1)	9(1)	-9(1)
N(9)	56(1)	39(1)	57(1)	-0(1)	22(1)	-12(1)
C(10)	47(1)	42(1)	48(1)	1(1)	10(1)	-5(1)
O(11)	50(1)	66(1)	70(1)	-10(1)	27(1)	-14(1)
C(12)	59(2)	63(2)	73(2)	-21(2)	11(1)	-5(1)
N(13)	37(1)	55(1)	47(1)	-9(1)	5(1)	-2(1)
O(14)	36(1)	78(1)	74(1)	-9(1)	15(1)	-10(1)
O(15)	52(1)	55(1)	64(1)	-2(1)	7(1)	10(1)
N(16)	34(1)	42(1)	41(1)	-1(1)	8(1)	2(1)
O(17)	56(1)	44(1)	52(1)	11(1)	4(1)	6(1)
O(18)	49(1)	56(1)	46(1)	-4(1)	-1(1)	-10(1)
N(19)	51(1)	45(1)	49(1)	3(1)	17(1)	-2(1)
O(20)	74(1)	72(1)	51(1)	-15(1)	19(1)	-18(1)
O(21)	58(1)	64(1)	67(1)	12(1)	29(1)	-7(1)
C(22)	59(2)	42(1)	48(1)	-3(1)	12(1)	-8(1)
O(23)	75(1)	55(1)	74(1)	5(1)	38(1)	-4(1)
C(24)	86(2)	47(2)	63(2)	-0(1)	16(2)	-1(2)
N(1')	52(1)	47(1)	60(1)	4(1)	29(1)	-2(1)
C(2')	56(1)	51(2)	43(1)	-1(1)	16(1)	2(1)
N(3')	37(1)	45(1)	51(1)	2(1)	14(1)	4(1)
C(4')	39(1)	44(1)	38(1)	-4(1)	11(1)	-1(1)
N(5')	41(1)	51(1)	33(1)	0(1)	4(1)	-3(1)
C(6')	36(1)	42(1)	44(1)	-3(1)	11(1)	4(1)
N(7')	46(1)	41(1)	48(1)	-3(1)	19(1)	2(1)
C(8')	42(1)	49(1)	56(1)	0(1)	11(1)	2(1)
N(9')	46(1)	41(1)	69(1)	1(1)	24(1)	3(1)
C(10')	43(1)	43(1)	60(2)	8(1)	14(1)	8(1)
O(11')	59(1)	47(1)	77(1)	-2(1)	36(1)	5(1)
C(12')	54(2)	44(2)	99(2)	2(2)	18(2)	3(1)
N(13')	41(1)	48(1)	57(1)	-9(1)	10(1)	3(1)
O(14')	44(1)	68(1)	90(1)	-15(1)	27(1)	-3(1)
O(15')	61(1)	76(2)	67(1)	10(1)	5(1)	22(1)
N(16')	35(1)	62(1)	37(1)	-8(1)	8(1)	-2(1)
O(17')	49(1)	80(1)	40(1)	14(1)	3(1)	-3(1)
C(18')	49(1)	67(1)	51(1)	-15(1)	8(1)	-11(1)
N(19')	50(1)	47(1)	42(1)	1(1)	13(1)	10(1)
O(20')	67(1)	52(1)	56(1)	-11(1)	15(1)	1(1)
O(21')	66(1)	68(1)	60(1)	2(1)	34(1)	7(1)
C(22')	41(1)	39(1)	85(2)	1(1)	16(1)	-1(1)
O(23')	63(1)	68(1)	122(2)	21(1)	53(1)	23(1)
C(24')	54(2)	46(2)	97(2)	10(2)	13(2)	-1(1)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$$

TABLE 5m Hydrogen coordinates ($\times 10^4$) and temperature factors ($\AA^2 \times 10^3$)

atom	x	y	z	U
H(1)	1381(33)	1185(17)	2566(30)	56
H(2a)	2377	1803	4175	54
H(2b)	4369	1674	3781	54
H(4a)	4486	2979	1800	45
H(4b)	5732	2433	2528	45
H(6a)	4825	4061	2315	47
H(6b)	5396	4262	3720	47
H(8a)	9866	4726	3316	58
H(8b)	8389	4732	4285	58
H(9)	8678(38)	5703(16)	2664(26)	54
H(12a)	1284	484	1049	77
H(12b)	3246	189	873	77
H(12c)	2395	757	-22	77
H(24a)	6289	6510	2192	76
H(24b)	7456	6755	3427	76
H(24c)	5291	6696	3376	76
H(1')	6635(33)	1357(17)	75(28)	61
H(2'a)	7877	2287	708	59
H(2'b)	9681	1999	219	59
H(4'a)	9153	2737	-2666	48
H(4'b)	10589	2378	-1687	48
H(6'a)	9498	3852	-3004	48
H(6'b)	10293	4318	-1896	48
H(8'a)	14697	4430	-2741	58
H(8'b)	13267	4779	-1952	58
H(9')	13903(36)	5150(18)	-4187(25)	60
H(12d)	5855	394	-977	78
H(12e)	7798	41	-787	78
H(12f)	6970	271	-2133	78
H(24d)	11745	6534	-4249	78
H(24e)	10539	6056	-5170	78
H(24f)	12708	6053	-5139	78

TABLE 1n Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
C(1)	164(10)	2834(11)	851(4)	60(3)*
O(2)	1709(8)	3174(8)	142(3)	77(2)*
C(3)	3659(11)	2927(11)	878(4)	61(3)*
C(4)	2085(9)	2447(9)	1688(4)	44(2)*
C(5)	1568(9)	49(9)	1973(4)	46(2)*
N(6)	510(7)	-422(7)	2862(3)	45(2)*
C(7)	1964(11)	-64(9)	3850(4)	57(3)*
C(8)	2906(11)	2350(10)	4210(4)	58(3)*
N(9)	4132(7)	3663(7)	3484(3)	46(2)*
C(10)	2788(9)	4127(8)	2570(4)	46(2)*
N(6a)	-1878(8)	-740(7)	2763(3)	53(2)*
O(6a)	-3023(7)	-790(7)	1931(3)	66(2)*
O(6b)	-2689(8)	-1014(7)	3542(3)	73(2)*
N(9a)	6474(8)	3855(7)	3526(4)	52(2)*
O(9a)	7321(7)	4492(7)	2790(3)	64(2)*
O(9b)	7564(8)	3444(7)	4303(3)	74(2)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2n Bond lengths (A)

C(1)-O(2)	1.433(8)	C(1)-C(4)	1.561(8)
O(2)-C(3)	1.457(8)	C(3)-C(4)	1.556(8)
C(4)-C(5)	1.542(8)	C(4)-C(10)	1.509(7)
C(5)-N(6)	1.463(7)	N(6)-C(7)	1.445(6)
N(6)-N(6a)	1.369(7)	C(7)-C(8)	1.511(8)
C(8)-N(9)	1.461(7)	N(9)-C(10)	1.454(7)
N(9)-N(9a)	1.367(7)	N(6a)-O(6a)	1.215(6)
N(6a)-O(6b)	1.242(7)	N(9a)-O(9a)	1.227(7)
N(9a)-O(9b)	1.226(7)		

TABLE 3n Bond angles (deg.)

O(2)-C(1)-C(4)	92.3(5)	C(1)-O(2)-C(3)	92.2(4)
O(2)-C(3)-C(4)	91.6(5)	C(1)-C(4)-C(3)	85.8(4)
C(1)-C(4)-C(5)	115.2(4)	C(3)-C(4)-C(5)	111.1(5)
C(1)-C(4)-C(10)	112.2(5)	C(3)-C(4)-C(10)	117.2(4)
C(5)-C(4)-C(10)	114.0(4)	C(4)-C(5)-N(6)	114.7(5)
C(5)-N(6)-C(7)	121.7(4)	C(5)-N(6)-N(6a)	117.8(4)
C(7)-N(6)-N(6a)	119.0(5)	N(6)-C(7)-C(8)	111.3(5)
C(7)-C(8)-N(9)	110.6(5)	C(8)-N(9)-C(10)	120.7(4)
C(8)-N(9)-N(9a)	117.5(5)	C(10)-N(9)-N(9a)	118.8(4)
C(4)-C(10)-N(9)	117.3(5)	N(6)-N(6a)-O(6a)	118.1(5)
N(6)-N(6a)-O(6b)	116.0(4)	O(6a)-N(6a)-O(6b)	125.8(5)
N(9)-N(9a)-O(9a)	116.8(5)	N(9)-N(9a)-O(9b)	117.5(5)
O(9a)-N(9a)-O(9b)	125.6(5)		

TABLE 4n Anisotropic temperature factors ($A^2 x10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	46(4)	77(4)	61(4)	27(3)	6(3)	25(4)
O(2)	81(3)	111(4)	54(2)	36(2)	22(2)	46(3)
C(3)	48(4)	81(4)	61(4)	27(3)	24(3)	21(4)
C(4)	31(3)	55(3)	48(3)	22(3)	8(3)	14(3)
C(5)	37(3)	54(3)	51(3)	9(3)	14(3)	17(3)
N(6)	32(2)	47(3)	51(3)	10(2)	9(2)	7(2)
C(7)	56(4)	59(4)	60(4)	22(3)	20(3)	19(3)
C(8)	50(4)	75(4)	49(3)	11(3)	9(3)	19(3)
N(9)	27(2)	50(3)	59(3)	7(2)	8(2)	9(2)
C(10)	29(3)	43(3)	68(3)	16(3)	10(3)	12(3)
N(6a)	44(3)	42(3)	71(3)	5(2)	18(3)	9(2)
O(6a)	36(2)	78(3)	83(3)	15(2)	10(2)	18(2)
O(6b)	66(3)	67(3)	92(3)	9(2)	51(3)	12(2)
N(9a)	36(3)	45(3)	73(3)	-4(3)	2(3)	15(2)
O(9a)	40(2)	69(3)	91(3)	14(2)	25(2)	22(2)
O(9b)	54(3)	81(3)	84(3)	2(2)	-14(2)	32(3)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$$

TABLE 5n Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
H(1a)	-279	4099	998	65
H(1b)	-1245	1569	671	65
H(3a)	4168	1717	707	66
H(3b)	5017	4243	1034	66
H(5a)	3042	-267	2085	49
H(5b)	483	-902	1423	49
H(7a)	1010	-867	4299	61
H(7b)	3282	-593	3839	61
H(8a)	4009	2550	4831	62
H(8b)	1603	2836	4306	62
H(10a)	1351	4285	2721	49
H(10b)	3745	5498	2372	49

TABLE 1p Atom coordinates ($\times 10^4$) and temperature factors ($A^2 \times 10^3$)

atom	x/a	y/b	z/c	U_{eq}
N(1)	-2263(3)	1336(2)	278(2)	58(1)*
N(2)	-2509(3)	2348(3)	-639(2)	66(1)*
N(3)	-851(4)	3165(3)	-682(3)	64(1)*
N(4)	690(2)	2549(2)	242(1)	47(1)*
C(5)	2961(5)	2972(3)	729(2)	55(1)*
C(6)	3541(5)	1810(4)	1747(2)	53(1)*
N(7)	1359(3)	1089(2)	1729(2)	53(1)*
C(8)	-211(3)	1579(2)	794(2)	42(1)*
N(9)	1066(3)	-64(2)	2464(2)	57(1)*
O(10)	-769(3)	-645(2)	2258(2)	74(1)*
O(11)	2622(3)	-374(2)	3224(2)	79(1)*
N(1x)	-1045(11)	-62(9)	1682(7)	57(4)
N(2x)	231(11)	-746(9)	2648(7)	70(4)
N(3x)	2252(11)	-203(9)	2903(7)	115(12)
N(4x)	2267(11)	878(9)	2066(7)	50(3)
C(5x)	3843(11)	1939(9)	1794(7)	83(16)
C(6x)	2505(11)	2834(9)	737(7)	122(17)
N(7x)	358(11)	1998(9)	515(7)	32(4)
C(8x)	296(11)	918(9)	1356(7)	39(3)
N(9x)	-1403(11)	2419(9)	-332(7)	52(4)
O(10x)	-3111(11)	1723(9)	-323(7)	96(7)
O(11x)	-1085(11)	3406(9)	-985(7)	105(10)

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

Table 2p Bond Length Å

N(1)-N(2)	1.366(3)	C(5)-C(6)	1.541(4)
N(1)-C(8)	1.305(2)	C(6)-N(7)	1.490(4)
N(2)-N(3)	1.313(3)	N(7)-C(8)	1.365(2)
N(3)-N(4)	1.356(3)	N(7)-N(9)	1.357(3)
N(4)-C(5)	1.481(3)	N(9)-O(10)	1.223(3)
N(4)-C(8)	1.322(3)	N(9)-O(11)	1.194(3)

Table 3p Bond Angles (deg.)

N(2)-N(1)-C(8)	103.3(2)	C(6)-N(7)-N(9)	123.2(2)
N(1)-N(2)-N(3)	112.7(2)	C(8)-N(7)-N(9)	124.4(2)
N(2)-N(3)-N(4)	104.0(2)	N(1)-C(8)-N(4)	111.3(2)
N(3)-N(4)-C(8)	108.6(2)	N(1)-C(8)-N(7)	141.0(2)
N(3)-N(4)-C(5)	135.9(2)	N(4)-C(8)-N(7)	107.6(2)
C(5)-N(4)-C(8)	115.4(2)	N(7)-N(9)-O(10)	115.3(2)
N(4)-C(5)-C(6)	103.0(2)	N(7)-N(9)-O(11)	116.0(2)
C(5)-C(6)-N(7)	101.7(2)	O(10)-N(9)-O(11)	128.7(2)
C(6)-N(7)-C(8)	111.9(2)		

*Bond distances and angles for the minor disorder form do not differ significantly from the major form.

TABLE 4p Anisotropic temperature factors ($A^2 x 10^3$)

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	40(1)	65(1)	66(1)	-9(1)	2(1)	-1(1)
N(2)	46(1)	86(1)	56(1)	-11(1)	-10(1)	14(1)
N(3)	56(1)	80(2)	50(1)	8(1)	-0(1)	16(1)
N(4)	42(1)	51(1)	45(1)	3(1)	7(1)	2(1)
C(5)	36(1)	68(2)	61(2)	10(1)	11(1)	1(1)
C(6)	38(1)	62(2)	54(2)	2(1)	1(1)	0(1)
N(7)	44(1)	56(1)	57(1)	14(1)	4(1)	-4(1)
C(8)	40(1)	42(1)	43(1)	-2(1)	8(1)	1(1)
N(9)	70(1)	50(1)	53(1)	2(1)	18(1)	4(1)
O(10)	83(1)	67(1)	75(1)	5(1)	26(1)	-22(1)
O(11)	86(1)	83(1)	65(1)	24(1)	7(1)	18(1)

The anisotropic temperature factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + \ldots + 2hka^*b^*U_{12})$$

TABLE 5p Hydrogen coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x	y	z	U
H(5a)	3743(32)	2745(27)	117(15)	70
H(5b)	3222(36)	4004(15)	1036(19)	70
H(6a)	4508(30)	1037(20)	1563(20)	62
H(6b)	4158(32)	2237(24)	2502(12)	62
H(5ax)	4291(11)	2591(9)	2486(7)	70
H(5bx)	5104(11)	1444(9)	1560(7)	70
H(6ax)	2260(11)	3851(9)	969(7)	70
H(6bx)	3103(11)	2837(9)	49(7)	70

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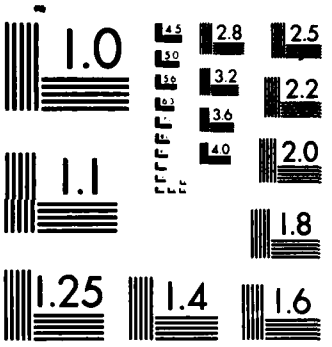

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